november, 1961

# nlgi spokesman

journal of the national lubricating grease institute

## Fluid Gear Lubricants – Their Future

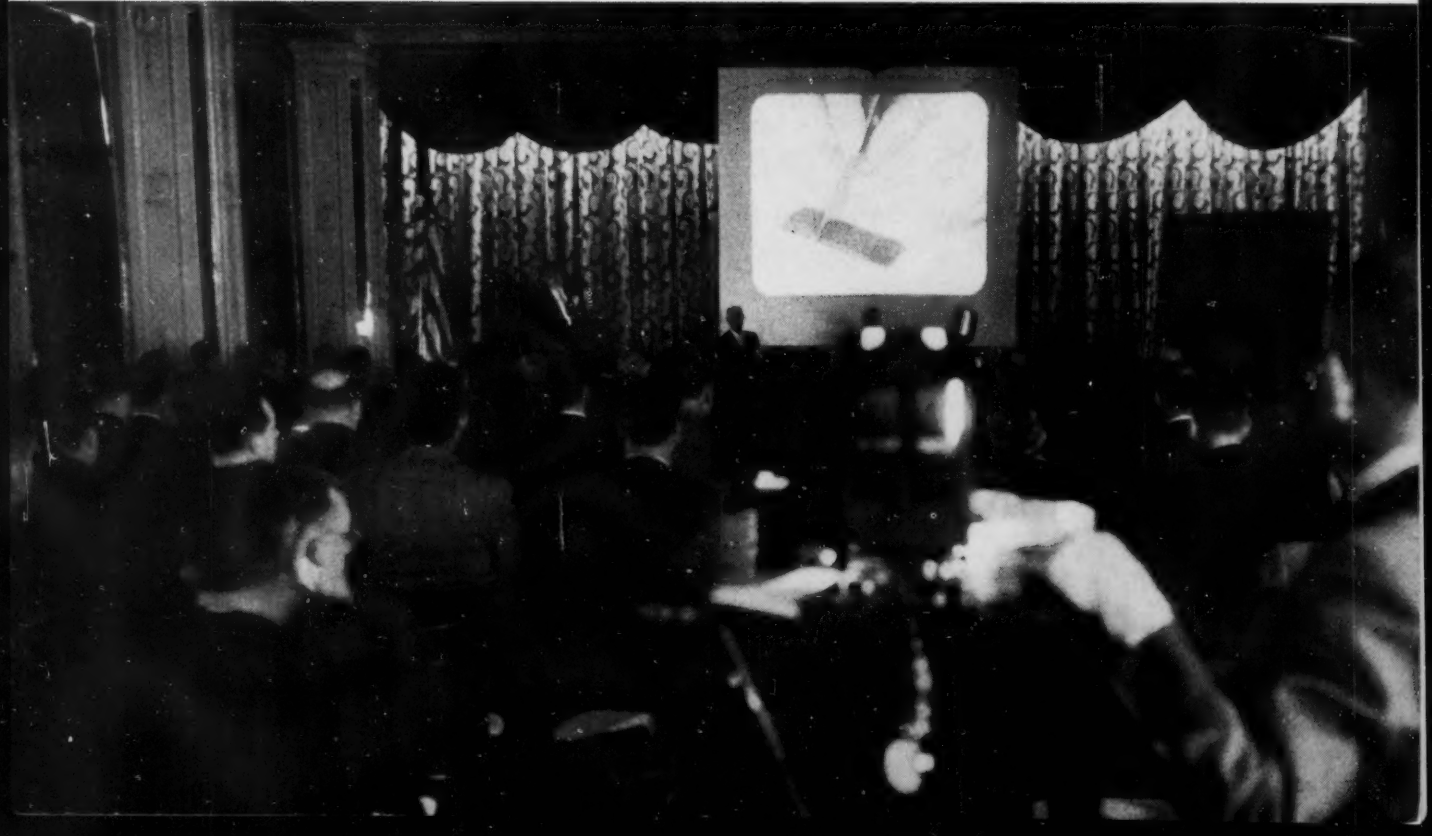
By H. J. KORP

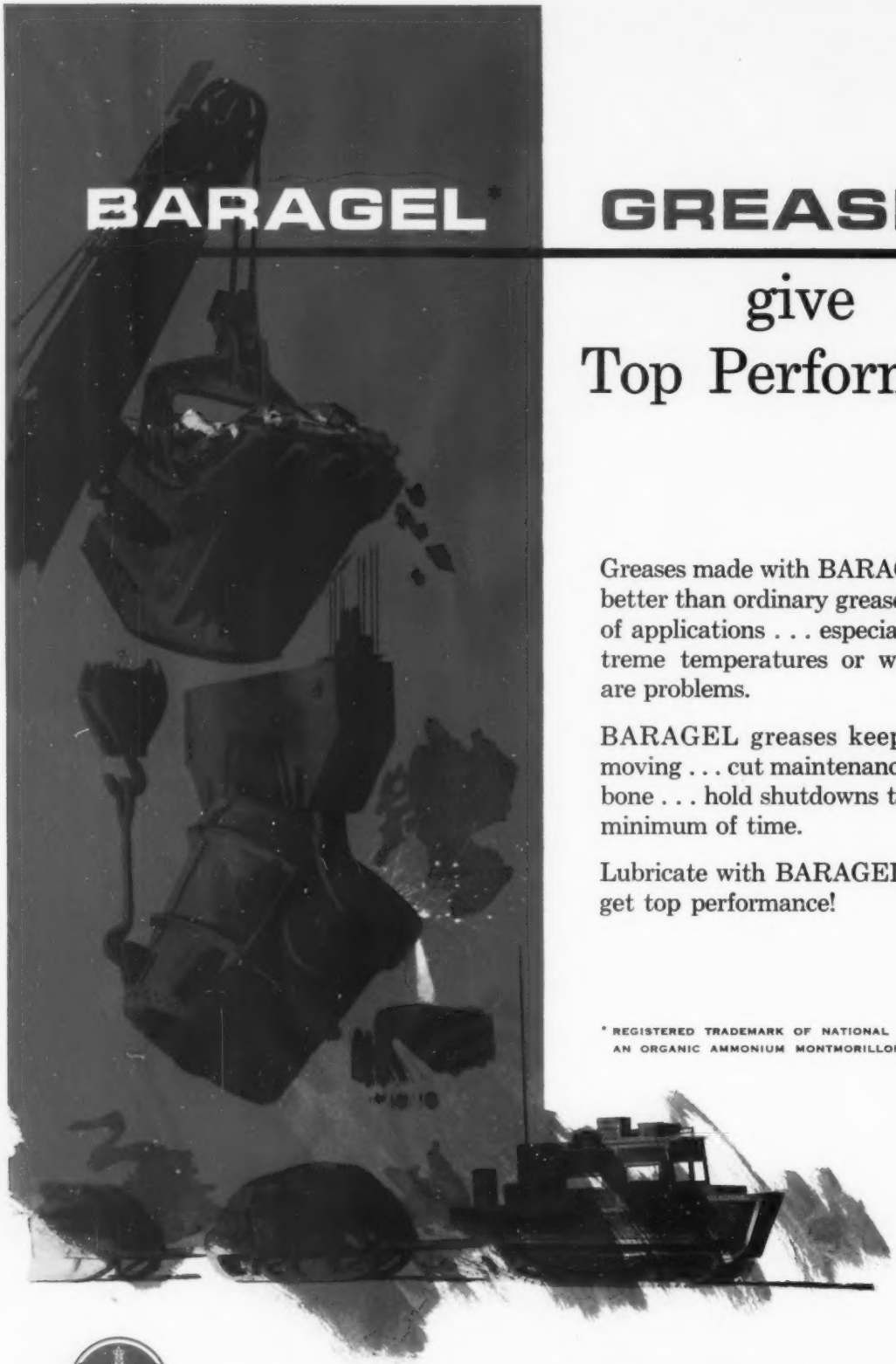
## Nature of Acetate Complexes in Greases

By J. PANZER

## Creativity Is Where You Find It

By H. L. HEMMINGWAY





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# nlgi spokesman

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## THE COVER

ANNUAL MEETINGS serve a great number of purposes . . . not the least of which is the opportunity to become acquainted with other men in the industry. But certainly an important adjunct of each yearly session is the exchange of data and information obtained at the meetings, while the papers are being given. The meetings represent the combined progress of the Institute, while the papers reflect the latest theories and practices. Beginning with this issue, the papers given at Houston October 29-November 1 will be brought to you in succeeding copies of your Institute journal.


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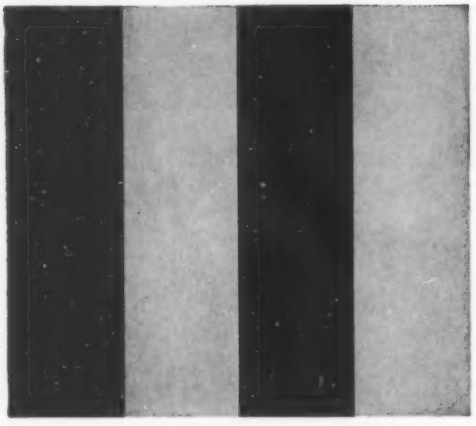


# NLGI PRESIDENT'S PAGE

*By* C. L. JOHNSON, *President*

# An Important Subject

Over the years my 28 predecessors have dealt with a number of important subjects on this page and in the twelve months to come, I will attempt to write on matters pertaining to the Institute and the industry in as effective a manner as the presidents before me.

I can think of no more vital subject to begin with than that of finances. Every member firm has had the problem of rising costs and shrinking profits to contend with, so it should not come as a surprise that inflation has caught up with NLGI at last. In spite of the utmost economy of operation, constantly reviewed by your Board of Directors, the Treasurer anticipates a deficit for the fiscal year 1961, and the cost trend continues upward.

Briefly, for your review, NLGI has maintained the same budget for over ten years, on a dues schedule established back in the 30's. Think of what your own costs have done in the past decade, let alone in the past 25 years.

The situation is not desperate but it does call for consideration by the membership. There are reserves to take care of this year's loss, but this money is also needed for a number of other purposes, and should not be drained away at an increasing rate. Part of the loss is caused by a falloff in advertising in the NLGI SPOKESMAN, a problem many journals have been facing. The directorate is aware of the need for more ads and a continuing campaign for increased lineage will be given even more emphasis in the administration to come. Services sold through the national office offer some income, but these are sold as close to cost as possible, as part of the benefits of membership, and do not represent an effort by NLGI to make a profit.

The basic source of income, then, is dues. Whereas this money once not only ran the Institute but allowed for some very nominal savings, in the past five years alone, your association has added: the Annual Production Survey, a second research fellowship at Northwestern University in addition to the existing program sponsored by NLGI at the University of Utah, a larger (and more expensive) Annual Meeting, a subsidized movie on lubricating greases, and the dynamic programs of the Chassis Lubrication Committee, just to name a few.

Your Board of Directors will continue to look for ways to reduce expenses and to increase income. In the year to come, a general review of dues payments by Active and Marketing member firms will be made to insure equitable charges to all. More members will be sought, and reevaluation of existing programs will be continued.

The financial situation does not mean the Institute's plans for growth will be slowed down, but as services expand we must continue to serve the much desired needs of the industry.

**THE PRODUCT:**

SANTOPOID® 23-RI
*field-proven* additive for
multipurpose gear oils

**THE RECORD:**

FIVE YEARS SERVICE ON SIX CONTINENTS

**THE REASONS:**

Santopoid 23-RI formulations meet the requirements of automobiles, trucks, heavy equipment, all types of military vehicles.

*Multi-Functional:* Requires only 2% by weight for mild EP; 5% for MIL-L-2105; 9.5% for MIL-L-2105B!

*Unexcelled:* For extreme pressure, anti-wear, lubricity, anti-rust, foam stability, and base stock compatibility.

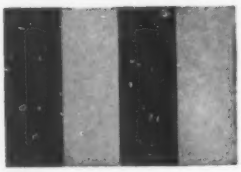
**THE USERS:**

Discerning gear oil producers
Hundreds of MIL-L-2105B approvals
Recommended or accepted by major manufacturers of automobiles, trucks, and earthmoving equipment.

**THE PROOF:**

In case you're not yet fortifying gear oils with SANTOPOID 23-RI, we'll be glad to arrange tests with your stocks—and send you literature containing convincing proof of performance.
*Write today on your letterhead to*

**MONSANTO CHEMICAL COMPANY**
Organic Chemicals Division
Dept. 4464, St. Louis 66, Mo.



# News About NLGI

## Names Representative

*British American Oil* of Toronto has named N. D. Morgan, national supervisor of product application, as NLGI Company representative, from this Active member firm.

## Prepare for Fifth NLGI Annual Production Survey

Production totals in pounds produced in the calendar year of 1961 will again be collected for the Institute, on lubricating greases and fluid gear lubricants.

The compiling agency is Ernst & Ernst, national certified public accountants. E&E have gathered the data since the inception of the survey, and in this way insure that replies are anonymous.

Company representatives of Active member firms in the U.S. and Canada will receive sample questionnaires in December, followed by actual questionnaires from E&E in January. One new question has been added, asking if the firm participated in previous surveys.

Survey results are given to members in all categories, in the spring.

## Joint Container

*The following, submitted by W. A. Magie, Magie Bros. Oil Co., chairman of the API-NLGI Joint Container Subcommittee, is a report of the Petroleum Packaging Committee meeting held recently in Chicago, Ill.*

### Shipping Cases

Nine companies are now using 24/1 gap cases, with many companies planning to start. One company plans export tests on this package.

*175# Test 24/1 Quart Case*

Approval will be sought to test the 175# 24/1 gap case, but opposition is expected since the rail classification people turned down a request to test 175# 24/1 fullflap cases not long ago.

### Large Container Committee

*New Lightweight 5-gallon Lug Cover Pail*

The subcommittee chairman attended a demonstration of this package at the Inland Steel Container plant in Jersey City in March. The results of the tests were so encouraging that arrangements were made for a special presentation of the development of this pail at this meeting in Chicago. This new pail has 29 gauge body and bottom with a 26 gauge top.

*Standardizing of the New Lightweight 5-gallon Lug Cover Pail and the 5-gallon Taper Pail*

The Large Container Committee and the Steel Shipping Container Institute will set up a joint committee to consider standards for either or both of the above pails.

*High Tensile Steel 20/18 Gauge Drum*

The use of high tensile steel in manufacturing 20/18 gauge drums may increase the strength sufficiently to promote wider acceptance of this package. The additional cost, however, may eliminate the incentive to use the 20 gauge package.

*Use of 120 Pound (16 gallon) 24 Gauge FRH Drums for Oils*

The subcommittee suggested that permits should be requested to test this package in shipments of lubricating oils lighter than 900 seconds Saybolt at 100° F.

*Bulk Handling*

One company is extending the use of rubber tanks for transfer of greases.

### Labels and Markings

The Federal Hazardous Substances Labeling Act was approved July 12, 1960. The subcommittee recommended that the members of the Petroleum Packaging Committee individually protest to the Department of Health, Education and Welfare to postpone the effective date from August 1, 1961 to February 1, 1962, and to request a hearing to consider the whole situation. However, August 12, 1961 was extended to February 1, 1962 for industrial products not toxic or highly inflammable.

### Traffic

*20/18 Gauge 55-gallon Drums*

The expiration date for the approved use of this package in truck shipments was extended August 1 indefinitely.

*20/18 Gauge 400 Pound FRH Drums*

Public hearings will be held in the fall by the Rail Classification Boards to decide on acceptance of this package for rail shipments of liquids.

### Petroleum Packaging Industry Advisory

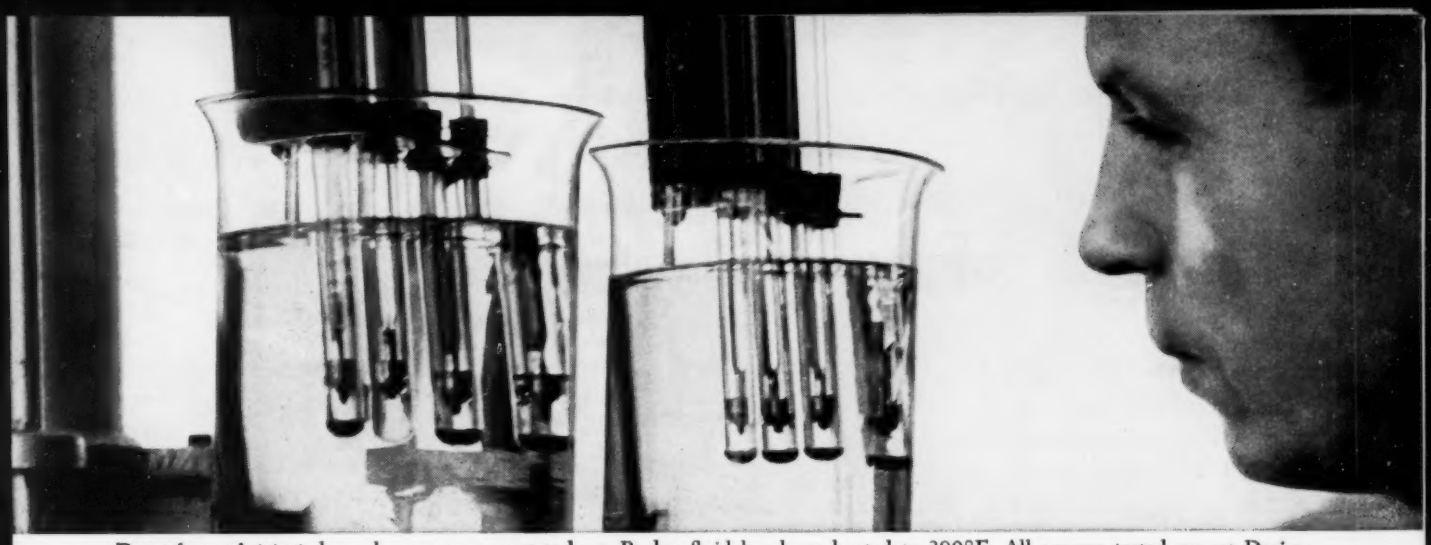
The Military Petroleum Supply Agency's Petroleum Packaging Industry Advisory Committee has recently been dissolved because its principal objectives have been fulfilled. However, the close working relationship between MPSA and the PPC will be continued.

### Small Containers

*Lightweight One-Quart Motor Oil Cans*

A survey shows that 21 companies have tested these cans to some degree, and that one company has been filling satisfactorily for two months. The most common weights of plate used so far are 60# bodies and 65# ends. Some companies re-

*Continued on page 262*



**Dropping point test** shows how greases react to heat. Beaker fluid has been heated to 390°F. All greases tested except Darina (second tube from left) have passed from solid to liquid state.

# BULLETIN:

## Shell reveals the remarkable new component in Darina Grease that helps it save up to 35% on grease and labor costs

**Darina® Grease is made with Microgel*, the new thickening agent developed by Shell Research.**

**Darina lubricates effectively at temperatures 100° hotter than most conventional soap base greases can withstand.**

**Read how this new multi-purpose industrial grease can help solve your lubricating problems and even save you up to 35% on grease and labor costs.**

THERE IS no soap in Darina Grease. No soap to melt away—wash away—or dissolve away.

Instead of soap, Darina uses Microgel—a grease component developed by Shell Research.

### What Microgel does

Because of Microgel, Darina has no melting point. It won't run out of gears or bearings.

Compared with most conventional soap-base greases, Darina provides significantly greater protection under adverse service conditions.

Mix water into Darina and the grease does not soften. It shrugs off water—won't emulsify.

### Resists heat

Darina will withstand operating temperatures 100° hotter than most conventional multi-purpose greases. It cuts leakage and reduces the need for special high-temperature greases.

Also, Darina resists slumping, thus forming a more effective seal against foreign matter.

### Saves money

Shell Darina can reduce maintenance expenses while it protects your machinery. Savings of up to 35% on grease and labor are quite possible.

In some cases lubrication intervals have been extended to double what they were before. Less grease is consumed and less time consumed applying it.

For details, see your Shell Representative. Or write: Shell Oil Company, 50 West 50th Street, New York 20, New York.

*Registered Trademark

SHELL

**A BULLETIN FROM SHELL**
**—where 1,997 scientists are helping to provide better products for industry**

# Future Meetings

**NOVEMBER, 1961**

6-7 Petroleum Packaging Committee, Packaging Institute, Fort Shelby Hotel, Detroit, Mich.

9-10 SAE National Fuels and Lubricants Meeting, Shamrock-Hilton Hotel, Houston, Texas.

13-15 American Petroleum Institute Annual Meeting, Conrad Hilton Hotel, Chicago.

26-Dec. 1 ASME Winter Annual Meeting, Statler Hilton Hotel, New York.

**JANUARY, 1962**

8-12 Society of Automotive Engineers, Annual Meeting, Cobo Hall, Detroit, Mich.

21-26 ASTM Committee D-2 meeting, Chase-Park Plaza Hotels, St. Louis, Mo.

**FEBRUARY, 1962**

21 NLGI Board of Directors meeting, Sheraton-Cadillac Hotel, Detroit, Mich.

**APRIL, 1962**

2-3 National Petroleum Refiners Association, Spring Meeting, Granada Hotel, San Antonio.

**MAY, 1962**

21-22 API Division of Marketing Lubrication Committee, meeting of subcommittee and technical program session, The Chanteclar Hotel, Ste. Adele-en-haut, Quebec, Canada.

22-25 API Division of Marketing, Midyear Meeting, Queen Elizabeth Hotel, Montreal, Canada.

**JUNE, 1962**

4-6 ASME Lubrication Symposium, Deauville Hotel, Miami Beach, Fla.

10-15 ASME Summer Annual Meeting, Hotel Frontenac, Quebec, Canada.

24-29 ASTM Annual Meeting and Apparatus Exhibition, Hotel Statler, New York City.

**OCTOBER, 1962**

16-18 ASME-ASLE Lubrication Conference, Pittsburgh Hilton Hotel, Pittsburgh, Pa.

21-24 NLGI Annual Meeting, Edgewater Beach Hotel, Chicago.

**NOVEMBER, 1962**

25-30 ASME Winter Annual Meeting, Statler Hilton Hotel, New York City.


**HARSHAW LEAD BASE**

Harshaw Lead Base, as an additive to petroleum lubricants, improves extreme pressure characteristics and imparts the following desirable properties:

- Increased film strength
- Increased lubricity
- Improved wetting of metal surfaces
- A strong bond between lubricant and metal surfaces
- Resistance to welding of metals at high temperatures
- Moisture resistance and inhibits corrosion

Harshaw Lead Bases are offered in three concentrations to suit your particular needs:

| Liquid 30% Pb | Liquid 33% Pb | Solid 36% Pb |
|---|---|---|

Other metallic soaps made to your specifications. Our Technical Staffs are available to help you adapt these products to your specific needs.

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1945 E. 97th Street • Cleveland 6, Ohio
*Branches In Principal Cities*



**QUALITY LUBRICANTS**

INDUSTRIAL
AND
AUTOMOTIVE

*Manufactured for*

REFINERS
COMPOUNDERS
JOBBERS

**FISKE BROTHERS REFINING CO.**

PLANTS
Newark, N. J. Toledo, Ohio

# NO GREASE STICKS LIKE JT-6

"Fifth wheels are the best proof of the ability of a grease to stay put," says Howard "Dink" Ray, head of Cato fleet maintenance. "And this is the first time we've ever had a fifth wheel come back off a long haul when the bare metal wasn't exposed!"

Mystik JT-6 is the finest multi-purpose grease ever tested by Cato—in the field or in the laboratory. The fact that it lasts longer, resulting in more time between lubrications, is due to a new raw material that has never before been used in the commercial production of a lubricating grease.

This unequalled ability to *stay put* on moving surfaces means a savings in time and maintenance costs. We believe there's no grease like it—and that once your customers have tried it they'll never accept a substitute. Write, wire or call collect for complete information.

**CATO OIL & GREASE COMPANY**
**OKLAHOMA CITY, OKLAHOMA**

CLIMAX MOLYBDENUM

# Molysulfide® News Digest

CLIMAX MOLYBDENUM COMPANY, a division of American Metal Climax, Inc., 1270 Avenue of the Americas, New York 20, N.Y.

# EDITORIAL PAGES OF LEADING TRADE MAGAZINES FEATURE $MoS_2$ INFORMATION

Higher temperatures, lower temperatures, greater pressures, longer lubrication life, all have combined to focus more attention on the uncommon lubricating values of molybdenum disulphide.

That's why editors are devoting more and more space to descriptions of what $MoS_2$ can and cannot do.

One article tells how $MoS_2$ prevents harmful friction in brake linings. Another discusses its use as a compressor lube substitute. Still another reports on tests of epoxy-resin compositions containing high concentrations of $MoS_2$ for industrial use on bearings, seals and friction strips.

Press fittings, wire drawing, cutting tools, ball joints—there are $MoS_2$ stories about these uses, too.

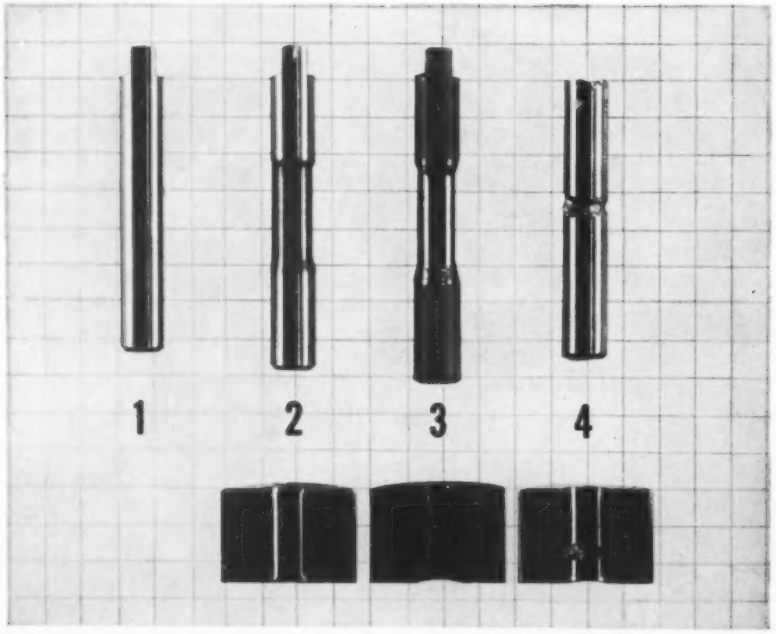
$MoS_2$ as a solid-film dry lubricant has been the subject of several articles. One details high-temperature properties; another describes a new bearing design that can increase bearing life as much as 450%.

Write us for a complete bibliography of published material on molybdenum disulphide.

*P.S.* Climax publishes a newsletter that describes even more uses. Let us know if you'd like your name on the mailing list.

*When writing, refer to CL-116*

## Proof of $MoS_2$'s High-Pressure Properties




Photograph above shows mild steel test pins. No. 1: Unused pin, No. 2 (lubricated with mineral oil and Molysulfide) and No. 3 (with Molysulfide bonded coating) were subjected to rotating pressures between bearing halves. Both were elongated and extruded without galling, seizing, or weight loss. No. 4 shows typical failure with conventional lubricant. Note that the key sheared off and pin and block were galled and seized.

*When writing, refer to CL-118*

## Use of $MoS_2$ Paste-Type Concentrates Shows Growth

These lubricants, containing higher-than-usual amounts of Molysulfide, first proved their value in press fit assemblies of pump gears and shafts. By completely preventing galling and seizing, and eliminating bent shafts, $MoS_2$ saved one company $1,821 annually on that operation alone.

The use of $MoS_2$ then spread to pins, engine block studs, and threaded connections, where not only ease of assembly but ease of disassembly is important.

Today, Molysulfide paste-type concentrates have proved to be the most versatile of $MoS_2$ lubricants. They are particularly effective wherever high load conditions exist—on splines, universal joints, ball joints, sleeve bearings, and machine ways and as wear-in and drawing lubricants.

*When writing, refer to CL-117*

# Fluid Gear Lubricants —Their Future

By: H. J. Korp, Southwest Research Institute

Presented as the Keynote Address at the
29th Annual Meeting of NLGI, Houston

MEMBERS of the National Lubricating Grease Institute and guests, it is a pleasure to be with you today and to be able to present to you some of my thoughts concerning the future of fluid gear lubricants.

As you must be aware, scientific research is perhaps the most dramatic and vital force affecting our way of life today. Technological advancements determine the success of our national defense, determine the state of our economy, and even determine the amount of leisure time available to each of us and, of course, how we can use it. Certainly on this basis, it can influence the future of fluid gear lubricants.

With regard to fluid lubricants, just as with every other area of technological activity, one has to continually take stock to see where he stands. Fluid lubricant requirements change. For example, there is much discussion of sealed systems, solid lubricants, gaseous lubricants, liquid metals and the use of anti-friction concepts to augment the load-support function of conventional bearings. I'm speaking of the use of such things as electromagnetism and electrostatic phenomenon in certain applications. All of these new considerations seem to indicate a poor future for conventional fluid products, but to me it simply affirms that in order to create or maintain a future for them the industry must objectively review the situation and make decisions to conduct scientific research programs as deemed necessary.

Research has a profound effect on industrial progress, and it may exhibit its effect in any of the following ways:

1. It may help create entirely new industries which never existed before.

2. It may revive and permit old industries to expand by lowering product production costs, improving present products, or by discovering desirable new materials, and

3. It may stimulate other industrial activity in support of new products of research.

It is hoped that the fluid gear lubricants industry is still in Category 2 but even if this is not so, supported research will permit the development of new industries as suggested in No. 1, above, to put to use the facilities, equipment and personnel of the industry as it presently exists. In recognizing the important role of science and technology in our industrial advancement, we can't overlook the fact that new industries very often displace or amend existing businesses. Cotton, for example, was affected by synthetics; railroads felt the influence of truck and air transport; the electric refrigerator replaced the iceman. It should be emphasized that in many cases the companies that manufactured the original displaced product were the ones that took over manufacturing and marketing of the new product.

Whether you like it or not, and it's wiser to like it, research and technology are going to give us fabulous new industries in the future. In any research development, there is considerable time lag – seven to ten years–between the beginning of research and the attainment of profitable production. The new things of this year were only research ideas in 1955 or earlier. Annual research expenditures have virtually doubled since 1955, therefore, we can expect increasing numbers of industries concerned with new products of research to come into being in the years immediately ahead of us.

Of course, it should be noted that research isn't the final step–research simply provides the ideas and the "know-how". The subsequent industrial development and marketing is done by other departments of our corporations and by individual businessmen. After research there must be extensive engineering and devel-

opment, investment of venture capital in new facilities, and finally, successful promotion and marketing.

But let me get back to fluid gear lubricants and try to show you why increasing research in this field is an absolute necessity. Today, fluid lubricants are required to perform under mechanical and environmental conditions unheard of just a few years ago. For example, let me tell you briefly about several of the problems that your industry might face in satisfying the lubrication requirements of aerospace vehicles now and in the near future.

The figures I quote are, of course, only to indicate the general nature of the problems. The magnitudes and relative order of importance are given in only an approximate and tentative manner.

Let's first talk about the pressure phenomena; or maybe it would be better to call it lack of pressure.

The pressure in the atmosphere varies from 760mm Hg on the earth's surface to about $10^{-18}$mm Hg in intergalactic space. High vacuum is known to be extremely detrimental to the proper functioning of conventional gears and bearings through loss of the lubricant or even the surface film of the lubricated parts due to evaporation or sublimation. Of course, because of the low oxygen content, there is a reduction in the formation of oxide films on the bearing surface, also. The metals and other materials used in gear and bearing construction themselves also evaporate or sublimate at high vacuum. In many cases, their mechanical properties, such as tensile strength, fatigue strength, etc., change rather unpredictably. All exposed gears and bearings for aerospace vehicles must be designed to cope with the effects of high vacuum. Where it is possible to seal them against outside pressure, it is generally advantageous to do so. The present design trend is to (1) partially seal the gears and bearings in the main propulsion system where the operating duration is short; (2) completely seal them in the secondary-power and cooling system where long life is desired; and (3) completely seal most of the instrument and control bearings where low friction and long life are demanded. Where control bearings cannot be hermetically sealed, novel bearings must be designed.

A second environmental condition to consider is temperature. The temperature in the ambient atmosphere is normally not an important design consideration, as there are overriding factors that are far more important. In enclosed gear and bearing systems, the operating temperature is determined by the heat dissipation due to friction within the system and the balance of heat transfer in and out. The following examples illustrate the range of temperatures experienced: In a propellant pumping system, bearings are necessarily exposed to the propellant and, in the case of cryogenic propellants, they will be at very low cryogenic temperatures during start up, as liquid oxygen boils at −298°F. Afterwards, however, these same bearings may be exposed to very high temperatures throughout the operational period. In secondary-power and cooling systems in aerospace vehicle application, liquid metals will probably be used as functional fluids. The liquid metal used is determined basically by the application, and the temperature may range from 1500 to 3,000°F or higher. The temperature for enclosed instrument gear and bearing systems depends in part on their location; in most cases, it is expected to be in the range of 500 to 1,000°F. The temperature of exposed bearings is determined by the energy balance; it is generally believed that during re-entry conditions, the temperatures may reach 3,000 to 5,000°F. For steady flight at very high altitudes, the temperature should be lower, perhaps in the neighborhood of 1,000° F. In control gears and bearings that are exposed to hot exhaust gases, the temperature may reach 4,000 to 5,000°F or more.

Then, we have the effect of solar radiation. This factor will mostly affect the novel bearings under consideration; heat radiation and charged particles will be the culprits. However, exposed cryogenic systems must undoubtedly be protected from direct solar heating, also. They should, in fact, be exposed to the "cold of space" on the shaded side of the space vehicle, if possible. The effect of solar heat on other bearings can be kept moderate by the proper choice of surface coatings. Effective techniques for controlling solar heating of satellites are already in common use. I previously mentioned the novel electrostatic bearings which might come into use; charged corpuscular radiation particles may affect such exposed bearings by partially neutralizing the charges. The effect, however, could probably be overcome by some shielding measure.

We also have the possibility of nuclear radiation. Nuclear energy sources will probably be used for secondary power systems in aerospace vehicles in the future. They present a serious problem to most conventionally lubricated gears and bearings, since the conventional lubricants are known to be affected seriously by nuclear radiation. Because of weight considerations, the use of full shielding cannot normally be tolerated. Unless conventional materials less affected by nuclear radiation are developed, novel gear and bearings systems not affected by nuclear radiation would need to be used. It should be noted, however, that all novel bearings are not free from radiation limitations. Radiation exposure does affect metallic core materials if electromagnetic type bearing approaches are involved. In addition, it is expected radiation will have adverse effects on insulatory materials and dielectrics used.

And, finally, acceleration and gravity effects must be considered. Space vehicle components are subjected to high acceleration, estimated at 10 to 60g's for a brief duration during launching. Further, zero gravity may

present serious problems in gear and bearing stability. Here again, it may require novel bearings to be designed to overcome the stability problem. For example, the stabilizing forces required can be built into such bearings by electrical or mechanical means.

Now, I realize I've gone far afield to present to you unusual environments which are difficult for your fluid gear lubricants to live with, but rest assured, they are the ones you will face in the not too distant future in more and more of your applications. Judging from what has happened with regard to aerospace accomplishments in the past few years, we should accept the fact that we will at least be approaching them. It can be expected that other more conventional lubrication requirements will also become more severe as new propulsion systems for more routine use come into being.

Of course, we all know that to meet these requirements, the performance capabilities of the products which comprise the fluid lubricants group—petroleum oils, synthetics and greases—are continually being boosted by the improved base stocks created by new refining techniques, and with the development and use of new additives. Much research along these lines has been accomplished, but much more can be done to improve properties of conventional oils and/or greases to perform under the expected more severe conditions.

However, because there are still shortcomings, research has developed synthetics, which will perform under conditions too severe for petroleum lubricants. These synthetics are usually manufactured from chemical raw materials other than hydrocarbons.

Although the new synthetics developed over the past decade are beginning to dominate the field of specialized lubricants, as you are aware, some researchers are taking a new look at highly refined or "superrefined" petroleum oils. These are conventionally refined products that have been treated by severe hydrogenation, acid treatment, or absorption to remove or reduce polar impurities, unsaturates and unstable hydrocarbon structures. These techniques improve oxidation and thermal stability and reduce corrosion and viscosity changes. This research is expected to produce new base stocks from petroleum oils to which improved additives can be added. This will permit us to produce lubricants having all of the properties of the best synthetics—but at a much lower price. This as I noted earlier is also a function of research.

Of course, other research goes on simultaneously to develop specialized materials which will provide lubrication at the super environmental conditions mentioned earlier. Approaches are considered to develop materials involving all three states of matter: gases, liquids and solids. Gas lubricated bearings have been developed and have been highly successful in some very high speed, low torque mechanisms. The gas selected need not have special properties, but must, for example, have non-corrosive properties insofar as the bearing materials are concerned.

Liquids have become very unconventional and are frequently metals of moderate melting point. Solids are used in what is termed "dry film lubrication," graphite and moly disulfides in an air atmosphere are effective to 700 to 750°F, inert gases such as nitrogen extend this temperature effectiveness further. More recently, various chemical compounds which can be bonded to bearing and gear surfaces directly with resins which withstand high temperatures have been suggested to act as lubricants, also.

Now, gentlemen, I really haven't told you anything about the future of the fluid gear lubricants industry except to point out that you had better keep your eyes and ears open to the changing technology and, whenever necessary, initiate and perform the necessary research programs. The long range outlook for business is for an economy "enormously larger and infinitely more complex than today's." Projections for 1975 for instance, include a gross national product of $1 trillion, 20 million additional homes, 30 million more motor vehicles on the highways, and as an absolute minimum, 1.1 million more scientists and engineers. Such estimates certainly indicate a tremendous future for your industry, you must be ready to take advantage of it.

In the technical papers that follow, I am sure we will all learn just what is being done to assure the future for fluid gear lubricants, and further, that the research which has been accomplished since your last meeting has already increased the potentials of your products. ■

## About the Author

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# Nature of Acetate Complexes In Greases

**By: J. Panzer**
**Esso Research & Engineering Co.**

THE TERM "complex soap grease" is generally used today to refer to those greases in which the thickener is made by mixing a soap with a low molecular weight salt or polar compound (such as acid or base)[1]. This class of greases has been further subdivided into two groups: (a) greases in which the soap and additives may be mixed in any proportion and whose "association is an adsorption behavior or a behavior similar to solubilization of polar compounds"[2]; (b) greases whose behavior suggests that the thickener is a chemical combination of soap and additive.

The literature indicates that greases made with soaps and salts of low molecular weight acids, particularly acetic acid, fall into the second grouping; i.e., that they are chemical complexes[1]. Because these greases have excellent lubricating properties, they have received considerable attention during the last few years. It was believed to be of some importance to elucidate the nature of the complexes to develop a better understanding of their effectiveness.

The results of our studies reveal that the earlier evidence supporting the concept of chemical complexes had been misinterpreted and that the soap-acetate thickeners appear to be more like the association complexes mentioned above, in which the soap is probably adsorbed on the surface of acetate particles.

The evidence which had been used in the past to establish these complexes as chemical compounds was of two kinds:

(1) When a mixture of acetate and soap in oil was heated to some specific temperature, the properties of the resulting grease differed quite markedly from those of a grease of the same composition in which the components were physically mixed at room temperature. Furthermore, the components of the room temperature mixture could be easily separated by solvent treatment while the thickener of the heated grease appeared to be completely homogeneous and could not be separated into multiple components except by chemical decomposition.

(2) X-ray diffraction patterns of the complexes were quite different from those of the soap and acetate used to make them and also were different from the published diffraction patterns of the other forms of the acetate. This was interpreted to mean that new crystalline species (not mixtures) had formed.

Reconsideration of the earlier data in our laboratory led to the speculation that the soap-acetate complexes might not be chemical compounds but adsorption complexes. If in the heating of a soap-acetate grease, the soap partially melts, becomes finely dispersed and is adsorbed on the surface of the acetate particles, it is understandable why such a system should behave differently from a simple mixture made at room temperature. Depending on how tightly bound the soap may be to the acetate, it may be difficult to distinguish such an adsorption phenomenon from compound formation. The heating process could also produce changes in the composition of the acetate itself or the soap itself that would account not only for the changes in the grease properties but also in the X-ray diffraction pattern. Yet the thickener in such a situation might be only a mixture and not a chemical complex.

Because we felt that it was desirable to understand more fully the behavior of the individual grease components before studying the complexes, our initial efforts were concerned with the changes in these components that might occur under the conditions encountered in making complex greases. This work, which was concerned with complexes of calcium and barium acetates, showed that the changes taking place in the X-ray diffraction patterns during complex formation could be accounted for entirely by changes in the acetates alone. Although it has not been established conclusively that the complexes are of the adsorption type, the behavior of the greases suggests strongly that they are.

The following discussion is concerned with the experimental support for the above conclusions. The calcium and barium complexes are discussed separately and experimental details are reported in the appendix.

### Calcium Acetate Complexes

There are several reports in the literature offering evidence which was interpreted as support for the concept that the thickeners in calcium soap-acetate greases may be chemical complexes. Amott and McClennan showed that, when calcium acetate and calcium oleate were heated in oil to 350°F., a grease was formed whose X-ray diffraction pattern apparently was quite different from those of calcium acetate and calcium oleate[2]. If the complex thickener were simply a mixture of acetate and soap, it would have been logical to assume that all the D-spacings calculated from the X-ray pattern of the complex could be attributed to the starting materials. However, Amott and McClennan found that several of the X-ray reflections produced by the acetate and oleate were absent in the pattern of the complex, while several new X-ray reflections had appeared. When the acetate and oleate were heated in toluene, a second complex with a new diffraction pattern was formed.

Still different complexes were formed when the soap-acetate mixture was heated above 400°F. Vamos and Guba[3] reviewed the patent literature and reported on their own observations of such complexes, the primary evidence being marked changes in grease properties above 400°F. Kolfenbach and Morway[4] also cited as evidence for the formation of complexes above 400°F. the changes in X-ray diffraction.

Before examining the complexes themselves we attempted to characterize the primary components—the calcium oleate and calcium acetate—as well as possible. A sample of calcium oleate was observed after heating to different temperatures ranging up to 450°F. Although the oleate softened during the heating process, its X-ray diffraction pattern after cooling did not change.

Calcium acetate behaves somewhat differently. It has been shown during the last few years that calcium acetate may form at least two hydrates, three anhydrous modifications, two chemical compounds with acetic acid and a compound with acetic acid and water, all of which have different X-ray diffraction patterns (one anhydrous form is amorphous)[5,6]. Our own work shows that calcium acetate monohydrate on being heated can form the half-hydrate and all three anhydrous forms, depending on the temperature[8].

Our examination of the published X-ray data for calcium soap-acetate complexes shows that every D-spacing can be accounted for by the presence of either the starting components or the hydrates, anhydrous forms and acetic acid compounds of calcium acetate. In Table I is shown the X-ray pattern of an oleate-acetate complex in oil made in our laboratory according to the procedure described by Amott and McClennan[2] (a Norelco diffractometer was used). Each D-spacing may be attributed to a specific compound which can be present in the grease.

**TABLE I**

**X-Ray Pattern of Calcium Oleate-Acetate Complex in Oil Heated to 350°F**

| *Principal D-Spacings in Å (Relative Intensity)*[1] | *Source Material* |
|---|---|
| 16.7 (VS)[2] | Calcium Acetate Half-Hydrate |
| 15.5 (W)[2] | Calcium Oleate |
| 11.7 (M) | Calcium Acetate Half-Hydrate |
| 8.6 (W) | Calcium Acetate Half-Hydrate |
| 8.4 (W) | Calcium Acetate Half-Hydrate |
| 7.5 (F) | Calcium Acetate Half-Hydrate |
| 5.57 (M)[2] | Calcium Acetate Half-Hydrate |
| 4.48 (M) | Calcium Oleate |
| 3.87 (F) | Calcium Acetate Half-Hydrate |
| 3.52 (F) | Calcium Acetate Half-Hydrate |
| 3.35 (W) | Calcium Oleate; Calcium Acetate Half-Hydrate |
| 3.28 (W)[2] | Calcium Acetate Half-Hydrate |

1. VS=very strong; S=strong; MS=moderately strong; M=moderate; W=weak; F=faint; VF=very faint.
2. Absent in Amott and McClennan pattern.

These X-ray measurements produced several reflections that were reported absent by Amott and McClennan, possibly because their observations were made with a powder camera, in which it is sometimes difficult to measure low angle reflections. The X-ray pattern of the anhydrous acetate used by Amott and McClennan as a reference in their study of the complexes seems to have been the pattern published in the ASTM X-ray Powder File which we now believe is incorrect. We feel that the more recently reported X-ray diffraction patterns of anhydrous calcium acetate are correct, because they were obtained with well-characterized material[5, 6, 8], in contrast to the pattern reported by the ASTM and obtained by Hanawalt with apparently uncharacterized calcium acetate[7].

The X-ray diffraction pattern which was obtained on a sample of Amott and McClennan "calcium complex 2" which they made by heating calcium oleate, calcium acetate, water and toluene, is shown in Table II along with the source compounds which account for each D-spacing.

**TABLE II**

**X-Ray Pattern of Calcium Oleate-Acetate Complex in Toluene Heated to 350°F**

| *Principal D-Spacings in Å (Relative Intensity)*[1] | *Source Material* |
|---|---|
| 12.7 (W) | Calcium acetate-acetic acid-water |
| 9.8 (M) | Calcium acetate-acetic acid |
| 8.23 (VF) | Calcium acetate-acetic acid-water |
| 4.44 (M) | Calcium oleate |
| 4.04 (F+) | Calcium acetate-acetic acid-water |
| 3.34 (F) | Calcium acetate-acetic acid-water |
| 3.17 (F) | Calcium oleate |
| 2.29 (VF) | Calcium acetate-acetic acid-water |
| 2.03 (F) | Calcium acetate-acetic acid-water |

1. Key to intensities in Table I.

The formation of the calcium acetate-acetic acid-water and the acetate-acetic acid compounds may be explained by the effect of unreacted oleic acid in the

calcium oleate. If any oleic acid were present, the calcium acetate would react to form free acetic acid which could then complex with the calcium acetate. The presence of excess acid in the finished complex (as reported by Amott and McClennan) seems to support the above hypothesis.

The X-ray patterns of the high temperature complexes can be accounted for by the presence of anhydrous calcium acetate. Table III summarizes our observations made with a complex of calcium oleate and calcium acetate heated to 420°F.

TABLE III

X-Ray Pattern of Calcium Oleate-Acetate Complex Heated to 420°F

| *Principal D-Spacings in Å (Relative Intensity)*[1] | *Source Material* |
|---|---|
| 15.5 (M) | Calcium oleate |
| 9.6 (S) | $\beta$ -anhydrous calcium acetate |
| 9.2 (M) | $\beta$ -anhydrous calcium acetate |
| 3.47 (W) | $\beta$ -anhydrous calcium acetate |
| 3.35 (W) | $\beta$ -anhydrous calcium acetate |

1. Key to intensities in Table 1.

Sometimes the D-spacings characteristic of the soap are absent in the X-ray pattern of the complex, especially if the complex is heated above 400°F. This phenomenon is probably due to the partial melting of the soaps and their fine dispersion in the grease, followed by adsorption onto the acetate particles. The small crystals thus formed can produce broad X-ray reflections of low intensity so that they may appear absent when compared to the reflections from the calcium acetate. We have observed this phenomenon with several different soaps. When the acetate and soap are mixed and heated in the dry state, no dispersion is obtained and the characteristic X-ray diffraction pattern of the soap is superimposed on that of the calcium acetate half-hydrate or $\beta$-anhydrous acetate depending on the temperature of heating.

Further evidence indicating that the calcium soaps and calcium acetate have not formed chemical compounds was obtained from observations of the complexes under different conditions. If the complexes were indeed chemical compounds, then changing the soap should produce changes in the X-ray diffraction pattern other than in those D-spacings characteristic of the soap. In actual fact, when other soaps, such as calcium caprylate, are used the X-ray pattern of the complex remains the same for a given method of preparing the grease. Changing the mole ratio of soap to acetate should change the crystalline structure of the complex if it is a chemical compound. Experiment shows that varying the mole ratio from 1:1 to 1:6 does not change the crystalline structure, but produces the same D-spacings from X-ray patterns.

There are some additional experimental observations which show that the behavior of the complexes, particularly the changes in the X-ray diffraction patterns, parallels the behavior of calcium acetate. The conversion of calcium acetate half-hydrate to the $\beta$-anhydrous form requires the elimination of water, usually by heating[8]. If a calcium soap-acetate complex made at 350°F. and containing the acetate half-hydrate is heated above 400°F. in the open, a new complex is formed whose X-ray pattern resembles that of the $\beta$ -anhydrous acetate. If the half-hydrate complex is heated, however, in a sealed container so that water cannot be eliminated, heating above 400°F. effects no change in the X-ray pattern.

Some of the experimental work strongly supports the suggestion that, although the complexes are not chemical compounds, there are strong interactions between the acetate and soaps. The $\beta$-anhydrous acetate in the isolated state rapidly absorbs water when exposed to air and reverts to the half-hydrate[6,8]. In the high temperature complex, however, the soap is apparently adsorbed on the acetate preventing it from picking up moisture readily. It is necessary to heat the high temperature grease with water in a sealed container in order to provide sufficient energy for the complex to dissociate and for the $\beta$-anhydrous acetate to react and form the half-hydrate.

The comparisons of the complexes with the behaviors of their individual components indicate that the complexes are neither chemical compounds nor simple mixtures, but are probably association complexes in which the soap is adsorbed on the acetate particles.

### Barium Acetate Complexes

Amott and McClennan reported the preparation of two barium acetate-oleate complexes whose X-ray diffraction patterns differ from those of the starting components and from each other[2]. One of these complexes was prepared by heating a mixture of barium acetate, oleic acid, barium hydroxide, oil and water; the other by heating a dispersion of barium oleate in oil with an aqueous solution of barium acetate.

As with the calcium complexes, our first step in studying the barium complexes was to observe the behavior of the barium soap and barium acetate separately. When barium oleate was heated to 450°F., no change in its X-ray diffraction pattern was found. A similar observation was made for barium acetate monohydrate. The possibilty that barium acetate might form complex compounds with acetic acid and water, by analogy with calcium acetate, was now investigated. When barium acetate monohydrate was treated with acetic acid, the X-ray pattern of the product was quite different from that of the monohydrate. Still another crystalline material, whose X-ray diffraction pattern was different from the other barium acetate salts, was obtained when the monohydrate was suspended in a small amount of water and filtered. A comparison of the X-ray patterns of these salts is shown in Table IV.

It should be pointed out that neither of the latter

two complex compounds was isolated in a pure state. They are mixtures in which the solvent (water or acetic acid) and probably some unreacted barium acetate monohydrate are present. Evidence for the presence of the monohydrate in these products is the fact that most of the D-spacings characteristic of the monohydrate are present in the X-ray diffraction patterns of the complex compounds. Table IV illustrates this quite clearly. The absence of some of the principal D-spacings of the monohydrate in the patterns of the complex compounds does not necessarily mean that the monohydrate is absent. It may be explained by orientation and crystallization effects which can diminish the intensity of some X-ray reflections so that they cannot be observed in the X-ray patterns. The essential point, however, is that the D-spacings which are not characteristic of the monohydrate must be due to new compounds involving reaction between the monohydrate and water or acetic acid.

**TABLE IV**

**X-Ray Diffraction Patterns of Barium Acetate**

Principal D-Spacings in Å (Relative Intensities)[1]

| *Monohydrate* | *Monohydrate + Water* | *Monohydrate + Acetic Acid* |
|---|---|---|
| 9.3 (VS) | 10.5 (VS) | 14.0 (VS) |
| 6.9 (S) | 9.6 (VS)* | 12.3 (VS) |
| 4.81 (W) | 6.35 (W) | 9.4 (M)* |
| 4.67 (M) | 5.28 (W) | 6.30 (M) |
| 4.39 (M) | 4.95 (W) | 4.80 (W)* |
| 3.68 (W) | 4.82 (W)* | 4.46 (W) |
| 3.52 (VS) | 4.20 (M) | 4.22 (W) |
| 3.45 (M) | 3.90 (W) | 3.60 (S) |
| 3.37 (S) | 3.70 (W)* | 3.46 (M)* |
| 3.28 (W) | 3.52 (VS)* | 3.17 (W) |
| 3.05 (M) | 3.42 (W)* | 3.10 (M) |
| 2.61 (W) | 3.37 (W)* | 2.89 (W) |
| 2.49 (M) | 3.28 (W)* | 2.62 (W)* |
| 2.40 (W) | 3.19 (M) | 2.54 (W) |
| 2.33 (W) | 3.04 (W)* | 2.50 (M)* |
| 2.21 (M) | 2.61 (M)* | 2.40 (W)* |
| 2.18 (W) | 2.49 (W)* | 2.02 (W)* |
| 2.07 (M) | 2.37 (M) | 1.93 (W)* |
| 2.00 (M) | 2.34 (W)* | |
| 1.93 (M) | 2.20 (W)* | |
| 1.73 (M) | 2.11 (M) | |
| 1.70 (W) | 2.06 (W)* | |
| | 2.00 (W)* | |
| | 1.93 (W)* | |
| | 1.83 (W) | |

1. Key to intensities in Table 1.
* Characteristic of monohydrate.

The D-spacings in the Amott and McClennan barium acetate-oleate complexes that could not be attributed to the acetate or soap now appear to be characteristic of these new crystalline species (See Tables V and VI).

It was observed that some of the most intense D-spacings characteristic of barium acetate monohydrate and its solvated compounds do not appear in the patterns of the complex greases, while some of the weaker lines do show up. This situation (which also appears with the solvated barium acetate monohydrate) may be explained by the following factors which are known to hinder characterization of crystalline materials especially in mixtures such as greases:

1. Orientation of thickener particles in the sample may lead to unequal exposure of all crystal planes to the X-ray beam so that some diffraction lines are diminished in intensity.

2. Crystallization may proceed preferentially along one axis to such an extent that along one or more axes the crystal may have too few planes to provide sharp, intense lines in the diffraction pattern.

3. Soaps sometimes do not crystallize completely in greases so that their X-ray reflections produce broad bands (especially at low angles) which may obscure diffraction lines characteristic of other substances.

The above described situation prevents positive iden-

**TABLE V**

**X-Ray Pattern of Barium Complex 1 (Amott and McClellan)[1]**

| *Principal D-Spacings in Å (Relative Intensities)*[2] | *Source Material* |
|---|---|
| 12.0 (W) | Barium acetate monohydrate + acetic acid |
| 9.11 (VF) | Barium oleate |
| 4.46 (VF) | Barium acetate monohydrate + acetic acid |
| 4.12 (M) | Barium oleate |
| 3.72 (MS) | Barium acetate monohydrate |
| 3.45 (F) | Barium acetate monohydrate |
| 3.24 (F) | Barium oleate |
| 2.90 (M) | Barium acetate monohydrate + acetic acid |
| 2.58 (W) | Barium acetate monohydrate + water |

1. Reference 2.
2. Key to intensities in Table 1.

**TABLE VI**

**X-Ray Pattern of Barium Complex 2 (Amott and McClellan)[1]**

| | |
|---|---|
| 13.4 (F+) | Barium acetate monohydrate + acetic acid |
| 4.18 (W)[3] | Barium acetate monohydrate + water |
| 3.93 (M—) | Barium acetate monohydrate + water |
| 3.72 (VF) | Barium acetate monohydrate + water |
| 3.07 (VF) | Barium acetate monohydrate |
| 2.88 (W) | Barium acetate monohydrate + acetic acid |
| 2.18 (VF) | Barium acetate monohydrate |

1. Reference 2.
2. Key to intensities in Table 1.
3. Although experimental error makes it difficult to attribute the 4.20 Å line of the monohydrate + water and the 4.22 Å line of monohydrate + acetic acid to different compounds, the methods of preparing these substances makes it likely that these lines are indeed different. The greater intensity of the 4.20 Å line and its nearness to the 4.18 Å line of the complex led to the assignment of the 4.18 Å line to monohydrate + water.

tification of the components of the barium complex greases. However, the probability that the soap and solvated compounds of barium acetate are responsible for all the diffraction lines is quite high in view of the observations made and because of the similarity between these systems and the calcium acetate greases which have been better characterized. The availability of excess water during the preparation of the complexes easily accounts for the formation of a hydrated barium acetate containing more than one mole of hydrated water. The presence of a barium acetate-acetic acid compound requires some explanation since no acetic acid was present initially during the preparation of the grease. A hypothesis which is consistent with the formation of such a compound is that the reaction between barium hydrate and oleic acid may be slow enough to permit some of the barium acetate to react with the oleic acid to form acetic acid which may now react chemically with the barium acetate.

The susceptibility of the barium acetate-soap dispersions to the conditions of preparation is further illustrated by using still a third procedure to make a complex. In this instance, barium acetate monohydrate and barium oleate (each prepared separately) were heated to 320°F. with a small amount of water in a naphthenic mineral oil. The X-ray diffraction pattern of the resulting smooth grease (in which the proportions of ingredients were the same as reported by Amott and McClennan for one of their greases) was different from those of the two earlier complex greases. Yet each D-spacing could be attributed to barium oleate or one of the compounds of barium acetate (see Table VII).

TABLE VII
X-Ray Pattern of Barium Complex 3
(Esso Research)

| *Principal D-Spacings in A (Relative Intensities)*[1] | *Source Material* |
|---|---|
| 10.2 (W) | Barium acetate monohydrate + water |
| 9.3 (VS) | Barium acetate monohydrate |
| 6.9 (M) | Barium acetate monohydrate |
| 4.57 (W) | Barium oleate |
| 4.39 (M) | Barium acetate monohydrate |
| 3.52 (S) | Barium acetate monohydrate |
| 3.37 (S) | Barium acetate monohydrate |
| 3.05 (S) | Barium acetate monohydrate |
| 2.61 (S) | Barium acetate monohydrate |

1. Key to intensities in Table 1.

By varying the conditions of making barium acetate greases, it should be possible to form a wide variety of products differing in composition.

There is some evidence showing that the barium acetate complexes are similar to the calcium complexes in that they are not simple mixtures. Amott and McClennan[2] showed that the solubility behavior of the complexes in toluene differed markedly from that of simple mixtures of barium soap and barium acetate. It was also demonstrated that separation of the complex into the components could not be effected by simple solvent treatment, whereas a mixture was separated easily.

### Conclusion

A study of calcium and barium complex soap-acetate greases has shown that the properties of these greases can be explained by the presence of mixtures in which the acetate and soap do not form chemical compounds. Much of the evidence suggests, however, that strong adsorption interactions between the soap and acetate develop during the preparation of the grease.

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## Appendix

Calcium acetate monohydrate, half-hydrate and three anhydrous forms were prepared and studied at different temperatures as earlier described[3]. X-ray diffraction patterns of greases and thickeners were obtained with a Norelco Diffractometer, using Cu-K $\alpha$ Radiation, $\lambda$ 1.542, and a nickel filter.

### I. Calcium Acetate Study

*Sample A.–Calcium Oleate*

A solution of 66 g. of USP oleic acid in 500 cc. of ethanol was added to a slurry of 7.8 g. of hydrated lime in 100 cc. of water. After stirring for a few minutes, 500 cc. of water were added and the mixture allowed to stand overnight. The calcium oleate was filtered and washed successively with ethanol, water, acetone and ether. Its X-ray pattern was obtained both before and after heating to 450° F. and found to be unchanged.

*Sample B.–Calcium Oleate-Calcium Acetate in Oil*

Calcium oleate-calcium acetate in oil was made according to the procedure of Amott and McClennan[2] using a naphthenic oil of 600 SUS viscosity at 100° F.

*Sample C.–High Mole Ratio Calcium Acetate-Soap Grease*

A mixture of 150 g. acetic acid and 75 g. $C_8$-$C_{12}$ fatty acids was added to a slurry of 125 g. hydrated lime in 925 g. naphthenic mineral oil (600 SUS viscosity at 100°F.) with stirring in a laboratory Hobart mixer. The mixture was heated to 350°F. and kept at this temperature for one hour after which a 100 g. sample was removed. The remainder of the grease was heated

to 430°F. at which temperature it was held for one hour.

The principal D-spacings (Å) in the X-ray diffraction patterns were as follows:

1. 350°F. product: 28.0, 16.7, 11.7, 8.6, 7.5
2. 430°F. product: 15.5, 9.6, 9.2, 3.47

*Sample D.—Low Mole Ratio Calcium Acetate-Soap Grease*

A mixture of 60 g. acetic acid and 175 g. $C_8$-$C_{12}$ fatty acids was added to a slurry of 75 g. hydrated lime in 700 g. naphthenic mineral oil as above. The mixture was treated in the same way as the high mole ratio grease. The principal D-spacings in the X-ray diffraction patterns were the same as observed for the high mole ratio products, but the intensity at 28.0 Å in the 350° grease was relatively greater (since a higher percentage of soap was present).

*Sample E.—Treatment of Isolated Thickener (Calcium Acetate Grease)*

A mixture of 90 g. acetic acid and 45 g. $C_8$-$C_{12}$ fatty acids was added to a slurry of 74.7 g. hydrated lime in 500 cc. n-pentane stirred in a 1000 cc. round-bottomed flask equipped with a Dean-Stark tube and reflux condenser. The mixture was refluxed while the acids were added so that water of reaction would be removed. When the addition was completed, refluxing was continued for one hour.

After cooling, a small sample of the resulting gel was exposed to air to evaporate the pentane. Part of the resulting powder was heated at 450°F. for 24 hours.

Some of the gel was placed in a 3 l. stainless steel Aminco bomb and heated for 24 hours at 450°F. and 500 psi pressure. After this time, the vapor was released to the atmosphere while keeping the temperature at 450°F. for an additional 24 hours.

The principal D-spacings (Å) in the X-ray diffraction patterns of the above products were as follows:

1. Gel: 28.0 + D-spacings of calcium acetate half-hydrate.
2. Thickener from gel: Same as gel.
3. Gel thickener heated to 450°F. in oven: 28.0 + D-spacings of beta-anhydrous calcium acetate.
4. Gel thickener heated to 450°F. in bomb: Same as calcium acetate half-hydrate.
5. No. 4 heated at 450°F. in oven: Same as beta-anhydrous calcium acetate.

*Sample F.—High Temperature Calcium Acetate "Complex" Plus Water*

200 g. of sample B and 50 g. water were stirred at room temperature in a Hobart kettle for 24 hours.

25 g. of sample B and 10 g. water were heated at 450°F. for 24 hours in a 300 cc. sealed bomb.

The X-ray diffraction patterns were as follows:

1. Room temperature reaction: no change.
2. High temperature reaction: D-spacings of soap and calcium acetate half-hydrate.

**II. Barium Acetate Study**

*Sample A.—Barium Acetate Monohydrate*

Barium acetate monohydrate was obtained from Baker Chemical Co. A sample heated to 450°F. showed no change in the X-ray diffraction pattern. A sample of the monohydrate wet with water was found to have a somewhat different X-ray diffraction pattern from the monohydrate. When a sample was wet with acetic acid, still another crystalline species resulted. A comparison of the three products is shown in Table VI.

*Sample B.—Barium Oleate*

Barium oleate was prepared by adding 56.5 g. USP oleic acid in 200 cc. isopropanol to 31.5 g. barium hydroxide octahydrate in 500 cc. water. The resulting precipitate was washed successively with isopropanol, water, acetone and di-ethyl ether followed by drying in air. Elemental analysis indicated that each mole of barium oleate was associated with 5 moles of water. The X-ray diffraction pattern was almost identical with that reported by Amott and McClennan[2]. No change in X-ray diffraction resulted after heating the barium oleate to 450°F.

*Sample C.—Barium Acetate-Soap Complex* 3

The "complex" grease was prepared by heating a mixture of 30 g. barium oleate, 160 g. barium acetate monohydrate, 60 g. water and 810 g. naphthenic mineral oil (600 SUS viscosity at 100°F.) in a Hobart kettle at 300°F. while stirring. Part of the resulting smooth grease was extracted with heptane to isolate the thickener which was essentially completely insoluble in water. The X-ray diffraction patterns of the grease and thickener were identical (see Table V). ■

## About the Author

J. PANZER *graduated with a BA degree from New York university and received his PhD degree from Cornell university in* 1956. *Since that time, he has been performing research on greases in the industrial lubricants and greases section of Esso Research & Engineering Co. Dr. Panzer is a member of the American Chemical Society. He has contributed to the* NLGI SPOKESMAN *several times in the past.*

# creativity is where you find it

By: H. L. Hemmingway, Pure Oil Company

*Presented at the NLGI 29th annual meeting in Houston, October, 1961*

IN YOUR company and mine, two areas of activity which may have the most profound influence on the company's future are marketing and research. Certainly, no other phase of modern business is unimportant, but spectacular success in either research or marketing can do much to insure the future of the enterprise. Unfortunately, the marketing function is frequently misunderstood and its importance under-rated by research people. And, just as often, the people in research, their method of operating, their aims and even their accomplishments may be misunderstood by marketing people.

During my own more than 25 years in the oil business, it has been my privilege to be closely associated with people in all segments of the business, but my closest associations have been with those in marketing and research. Strangely enough, in spite of the sometime mutual misunderstanding and the more obvious differences between researchers and marketers, there are also remarkable similarities. In the hope that this may promote better understanding of each by the other—and of both professions by all others—let us examine some of the similarities.

First, let me explain that these comparisons are made with some fear that when finished I will have fewer friends in both groups. While both are honorable professions, it is quite certain that many research people will be offended at being compared with "peddlers" and, likewise, many salesmen will cringe at any suggestion of similarity to the "long hairs" in research. Nevertheless, I am struck by the similarities in top-flight sales and research people more so than by their differences.

Let me explain also that this discussion applies primarily to "working" salesmen and research people. By "working" salesman, I mean the man who, by his own direct actions, influences the decision of the purchaser. The "working" research man is considered here to be one whose principal activity is research at the bench or at his desk. The fact that sales managers, administrators, technical service personnel, sales promotion people, advertising men, research directors, research administrators and others whose contact with actual selling or research is somewhat indirect are omitted from this discussion is *not* because their role is unimportant or that they lack creativity. They were omitted partly for simplicity and partly to emphasize that in either research or sales, the success of the endeavor is primarily depending on the "working" research man or salesman—and to a lesser extent on what takes place elsewhere in the hive.

Finally, let me make it clear that I discuss these matters not as a "working" salesman or research man, but simply as one having had the fortunate privilege of closely observing good men in both fields over many years.

I believe research and sales people are alike in these ways:

1. Both are ingenious individualists.
2. Both tend to be "lone wolf" workers.
3. Both work at jobs that are essentially "intellectual."
4. Both work at their jobs more than just during "working hours."

5. Both have a special need for recognition of their work.

6. Both have a tendency to gripe about how the company is being run.

7. Both may have similar problems with the usual type of promotion.

They are different in these ways:

1. The "values" that each considers important are quite different.

2. Training requirements are different.

3. Each has a sense of humor, but the kinds of humor differ.

Let us examine the similarities:

1. Most top-flight salesmen and many of the best research people are ingenious individualists.

True, many salesmen and research people conform, but the best in each field are more likely, it seems to me, to be strongly individual.

The individuality of the man may possibly express itself in dress, in the decor of his home or office, in his hobbies or in the car he drives; but these are, perhaps, only superficial expressions of individuality. More important is the individuality of the way the man thinks, especially in the solution of a problem or the development of a new idea. While ingenuity, creativity or individuality are expected of research people, it seems to me that not enough credit is given to such characteristics of a good salesman.

Let me illustrate with an example. A group of oil company people were attending a meeting at the factory of their tire supplier. The TBA manager had to leave the meeting early to return to the oil company home office for a very important meeting. The tire company account executive, a very able salesman, drove our TBA manager to the airport in a car belonging to one of the other tire company people because the TBA manager's brief case and baggage were in that car.

When they arrived at the airport, with only a few minutes to spare, they found that they did not have the key to the trunk. All of the papers for the important home office meeting were in the TBA manager's brief case in the locked trunk. This was the only connection he could make to arrive in time for his meeting, so our TBA manager was forced to board the plane sans brief case and baggage.

Imagine his surprise, when he arrived at the home office airport, to be greeted at the foot of the stairs from the airplane by the passenger agent who said, "Mr. S., I believe this is the case that you need for your meeting."

How was it done? First, of course, was the refusal to believe that it couldn't be done. The plane on which our TBA manager left was a local, and it made a leisurely stop or two before arriving at the town where planes had to be changed for the last leg of the trip. This provided time to get the missing key, charter a plane to the airport where the change in planes took place, get the important case on board just before the baggage compartment was closed, and then make a phone call to the passenger agent at the final destination.

You may say that this was no great exercise in ingenuity, but the solution of the problem was individualistic because I suspect that most of us would have been inclined to say, "It can't be done," and let it go at that.

2. Both tend to be "lone wolf" workers.

It is unlikely that there will be disagreement that research men tend to be "lone wolf" workers. A missile physicist is quoted[1] as saying, "The factors which help a creative scientist produce are hard to define because they seem to reduce so quickly to the statement he makes so often—'just leave me alone.' "

Perhaps the idea of the top-flight salesman being a "lone wolf" is more controversial. However, with sales teams, just as with research teams, it is usually one man who makes the sales breakthrough. The salesman tends to be nervous when he senses that the group calling on the purchasing agent is too large. He may ask for "technical assistance" in making the sale, but he strives to retain control of the sales situation.

In research, the team approach is frequently used. The new ideas coming from such teams are usually the products of individuals. The combination of two or more top-flight research men working on the same project, however, has many of the same problems of pride and rivalry as are generated when two top-flight salesmen share the responsibility for selling the same account.

Before leaving the subject of "lone wolf" effort, something should be said about aspects of both jobs which are essentially lonely.

Consider the salesman on his first call on an important prospective customer represented by a tough purchasing agent. With no one to help him, relying only on his wits, the future of his company to some extent depending on him, it is no wonder that he may feel lonely. In fact, this situation is such that some experts[2] have recommended dividing the sales force into two groups; one, those who have special abilities and can stand the wear and tear of introductory or "cold turkey" calls, and the other composed of those who prefer the "sustaining" type of sales call.

Likewise, the researcher whose mind is working in an area previously unexplored by any man may also feel lonely. But, in the case of *top-flight* salesmen *or* researchers, the excitement and challenge of these pioneering situations seems to more than compensate for the potential loneliness.

3. Both work at jobs that are essentially intellectual.

Certainly this is true in research, but some may argue that selling is hardly an intellectual pursuit. Webster defines *intellectual* as "Belonging or relating to, or performed by, the intellect or understanding." Creative selling would seem to be "performed by the intellect or understanding." While effective selling may not require the depth of study and analysis as research, most will agree that it requires a quick mind, knowledge of product, the quality of perception and the maturity that comes with experience.

4. Both work at their jobs more than just during working hours.

The literature is full of references to the research worker who finds regular working hours onerous. He may rebel at strict regulation of his time; he is certain to react violently to the installation of time clocks.

The matter of strict regulation of working hours for research personnel receives much management attention. Yet, a company employing 100 professional research workers may also employ several thousand salesmen. In most cases, the salesmen's boss doesn't know when his men get to work, how often they stop for refreshment, how long they take for lunch or how early they get home. Yet in the same organization, the hours of the research worker are likely to be strictly regulated.

In neither case is it easy to separate "working hours" from other hours. Ask the wives of research men or salesmen. What is the conversation likely to be at a party if a research man meets another—or a salesman runs into another salesman in the same line? During "non-working" hours, or even on vacation, does either—especially if he's good in his field—ever completely shed the mantle of his job?

At our Research Center, I may see more of one of our senior scientists when he's on vacation than when he is "working." He comes around frequently on his vacation, but as evidence of greater freedom and relaxation on "vacation" he may roam the laboratories more widely than when he is "working" and staying closer to his desk.

I think it is safe to say that, except for the time when he is in deep sleep, either the top salesman or research man is "working," however subconscious the effort may be.

5. Both have a special need for recognition of their work.

This is not to say that there isn't a need for recognition in almost any kind of work, but it seems to be especially strong in the case of salesmen or research workers.

I suspect that all good sales managers must set aside some portion of their time to reassure top salesmen and "hold their hands." Most researchers hunger for recognition also, although, as we shall see when we discuss the differences in the values or loyalties of researchers and salesmen, it may be of a somewhat different type than that which satisfies the salesman.

It is possible that, in the industrial situation, this is a natural result of the tendency to integrate what is essentially an individual achievement into the group or department record. In fact, I remember one salesman friend who frequently brought his orders directly to the home office, rather than to have them sent through the usual channels, just to make sure that they were properly credited.

6. Both have a tendency to gripe about how the company is being run.

Certainly neither researchers nor salesmen have a monopoly on second-guessing company management, but it is not surprising that aggressive, creative and intelligent people feel fully qualified to find fault in areas where their interest is intense. This should not be too disturbing to management, nor should it be ignored. Probably, it represents a perfectly normal, healthy situation.

Certainly, since it is the responsibility of research workers *not* to be satisfied with things as they are, there is little reason to believe that this attitude will apply only to research projects and not to other matters. (After reading this, another salesman friend pointed out that it was also a tendency for aggressive salesmen *not* to be satisfied with things as they are.)

7. Both may have problems with the usual type of promotion.

In many research laboratories, there are separate ladders of promotion. In addition to the normal progression via the administrative route, there is a parallel scientific ladder having equivalent salary and prestige at each step. Thus, it is possible for outstanding research scientists to progress as rapidly as their ability permits and to earn as much as the head of the laboratory. In some companies, I am told that it is theoretically possible for a research scientist to approach the earning power of the company president.

These separate ladder progression systems have come about for several reasons. First, many top research people have no interest in administration. Secondly, in some cases they lack aptitude for administration. Finally, and perhaps most important from the company viewpoint, when they become administrators, their research creativity slows down or stops.

For the top salesman, however, the normal progression as a reward for his efforts is via the administrative route. At first he is flattered at the promotion, and he needs the extra money. The paperwork probably bores him, he becomes restive at the "politics" of administration, and soon he may long for the action of selling. I think you will agree that a not infrequent result is the gain of a poor administrator and the loss of a good salesman.

Hopeful signs are indicated by the following quotations from a recent publication[3]:

"But," this executive continued, "over the long run, it's just possible that the scientist is not so different; that the same things which make our professional, high talent people more creative are also going to be effective in motivating everyone else; that all employees can at least be more productive, if not creative. In other words, the scientist may just be in a position where management is forced to pay more attention to his feelings, his likes and dislikes."

And from another part of this same report: "A few executives mention the importance of keeping promotion channels open, both within research and into other functions, such as marketing and manufacturing. More are concerned with keeping their skilled scientists doing what they were trained to do. Because, in the past salary increases over a certain level were only possible if the research man was moved to an administrative job, several companies say they have set up separate technical ladders of advancement for scientific people."

It seems to me that the separate ladder or promotion approach has considerable justification for the top salesman—just as much as for the top research man. And, there may be other jobs within oil companies where the same problems exist.

While there are these remarkable similarities between these two occupations, the differences are so obvious that in a casual meeting a perceptive individual would be unlikely to mistake a research man for a salesman or vice versa. In fact, Dr. Vannevar Bush is supposed to have said, "Research workers are no better or worse than other people—just different."

Some of the more obvious differences between researchers and salesmen are:

1. The values that each considers important are quite different.

The salesman's loyalty is strongly oriented toward the company and its products. While he may gripe to his fellow employees, he is strongly defensive to outsiders. Also, in the accomplishment of his work, primarily he seeks the accolades of his peers and superiors within his own company.

The researcher's values or loyalties are more likely to be divided between the company and his field of science. Frequently, he is more concerned about the accolades of his fellow scientists—in industry and the universities—than those of his fellow employees. It may be as important to him to present a creditable paper before a scientific society as it is to invent something of great value to his own company.

2. Training requirements are different.

Some of the best salesmen I have known either never attended college or got through "just barely." Most top research men today not only have Masters' or Ph.D. degrees, but were close to the top of their undergraduate classes. While the training of the top salesman is less formal, he has probably served his time in the "hard knocks" school.

3. Each has a sense of humor, but the kinds of humor differ.

The humor of the salesman is legend. It is inclined to be robust, heavy, earthy and obvious. The humor of the researcher is inclined to be devious, puckish and light.

Consider for example the sign on a cabinet in the laboratory of a lady researcher: "PLEASE USE THE HINGES!"

What a nice way to say "Shut the door!"

Or, the time when we were having a meeting to discuss a downward trend in our invention disclosures. When I came to the meeting room, at my place was a cartoon. It depicted a research laboratory with the director standing at the door saying, "Well, don't just stand there—invent something!"

On another occasion we had been discussing upward communication and the desirability of getting gripes out in the open, even though it was disagreeable. A few days later I received a cartoon in the office mail. A Caspar Milquetoast type was addressing a meeting. The caption: "Well, here's my opinion for what it's worth—my job, probably!"

Or, after we had had some down-to-earth discussions with some fairly sharp exchanges as to why even research people ought to be loyal purchasers of the company's products, and I stuck my head in the door of a small laboratory, without interrupting his work, the occupant of the laboratory said, "Well, hello—rabble-rouser."

Of course, there are many other differences between research and sales people, but no good purpose would be served by calling attention to them. What has impressed me is that there are so many similarities and, because of this, I believe that better mutual understanding is possible.

From my experience in both groups, here are a few simple suggestions which may help make the combination of research and sales within any company more effective:

1. Each should cultivate better understanding and tolerance of the other field.

This is directed more to researchers than to salesmen, but it applies to both. Some research people are inclined to view the salesman as a rather shallow fellow, given to stretching the truth and traveling in high style on a fancy expense account. To researchers having that attitude, I say, respect this man; he has a function in industry that is as essential to the success of the company as yours. Don't let the fact that he is "wired up" quite differently than you are cause dis-

respect. Instead, concentrate on how the things you have to offer can supplement and complement his needs.

And, to my friends in marketing, respect your research people and don't be annoyed because they may not consider selling as complex and full of hardship as you do. Also, respect their tendency to be conservative and to view with equal importance both the good and the bad points of the new product. Treat them as the competent specialists they are; not as academically-minded servants. And, above all, remember that they resent being called "long hairs" just as much as you resent being called "peddlers."

2. Let your research people know what your objectives and your problems are.

Give your research department as comprehensive and complete information on your marketing problems and objectives as possible, so that they may integrate their activities into your business plans. This should be much more effective than just telling research what to do without the reasons why and, above all, it beats trying to tell them how to do it.

It seems logical that effective action will result when both groups are working toward the same objectives—each in his own way.

3. Don't use your research people as scapegoats or just for complaints.

Sometimes, because the research group is out in the country somewhere, the temptation is great to use them as scapegoats when something goes wrong. Research should, of course, be held accountable, but should also be permitted to defend itself instead of being condemned in absentia.

Research people can and will help in the handling of major complaints, but this should be only a minor function of research.

4. Expose each group to the work of the other.

I have always been a firm believer in the merit of having a new salesman spend some time in the research laboratories as part of his training. From this he gains some understanding of and respect for what goes on in research, plus product knowledge. Also, he can speak to customers about his company's research position with more authority. It is also helpful to research people if they can sit in on sales meetings, actually participate in new product introductions, or in other ways be exposed to the sales atmosphere.

5. Plan ahead.

Most research programs involving new or improved products take time. As a general rule, completely new products take from three to seven years; sometimes even longer. Crash programs are expensive, inefficient, and probably take away from the overall creativity of the organization.

Anticipation of future requirements reduces the need for crash programs and provides the time required for an orderly development program.

Likewise, research people should not expect that a completely new product, just introduced to the marketing group, can be readied for the market in a few weeks. Effective marketing also requires time and effort.

While we have given the name "research" to those activities in which marketing and research people most frequently collaborate, many would classify them as more "product development" than research. Whatever the name, the point is that ultimate success is highly dependent on the most careful planning and coordination.

And now, you may wonder what has been gained by this discussion of research workers and salesmen. As mentioned at the beginning, I am convinced personally that in these two professions there resides the greatest potential to influence the future of your company and mine. If this has helped you to better understand either group, and through better understanding to more fully encourage and utilize this potential, then my time has been well spent.

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## About the Author


H. L. Hemmingway *received his Master's Degree from the University of Wisconsin in* 1932 *and, after a period as a high school science teacher, joined the Ashland Refining Co. in* 1933. *In* 1935 *he became associated with Kendall Refining Co. Since* 1944 *he has been with Pure Oil Co. where, in* 1957, *he was appointed director of research at the company's research center in Crystal Lake, Ill. Mr. Hemmingway was a member of the NLGI Technical Committee for several years and headed the subcommittee which originally developed the NLGI "Recommended Practices for Lubricating Automotive Front Wheel Bearings." He served as president of the Institute in* 1955 *and was a member of the board of directors* 1943-1944 *and* 1948-1958. *Mr. Hemmingway has contributed to the* NLGI Spokesman *before.*

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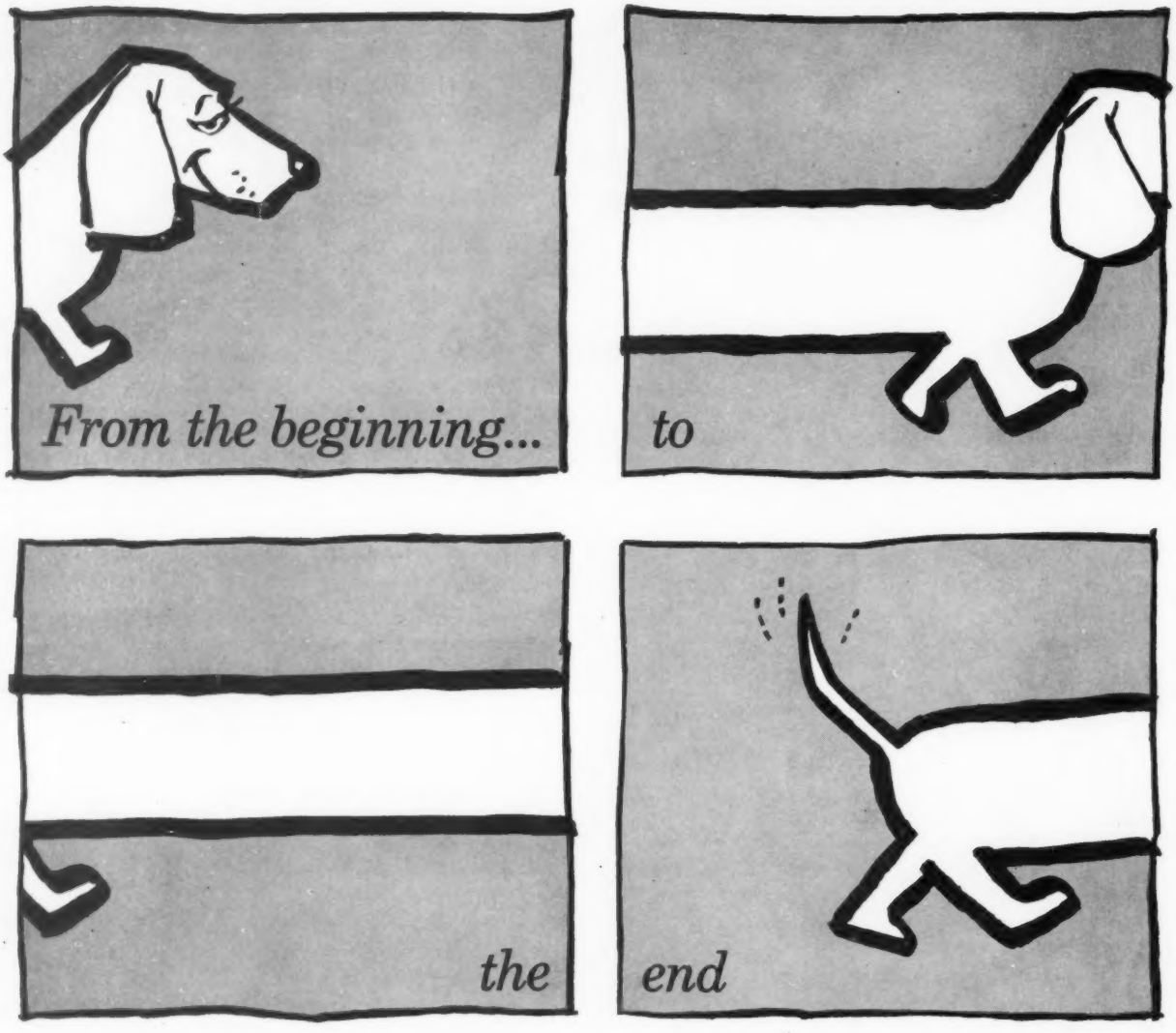


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# Literature and Patent Abstracts

## Structure

### Structure of Calcium Soap Fibers

R. J. Bird and G. Rooney, Nature 190, No. 4773, 337-8 (1961).

Soap fibers from a calcium base lubricating grease were examined under an electron microscope. A smear of the lubricant, which had been made from commercial fatty acids, was placed on a slide and washed with n-heptane before the observations. The fatty acids consisted largely of $C_{18}$ chain length some of which were unsaturated.

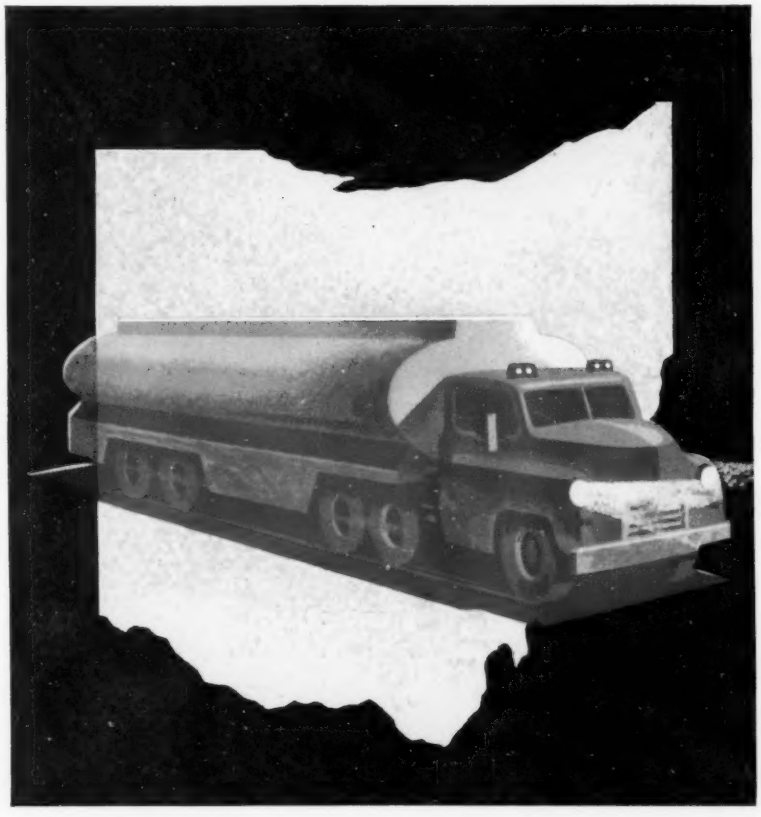
Both small twisted fibers of about 0.06 microns width, and ribbons of variable larger dimensions were observed. Both types had striations running parallel to the lengths and with spacings of about 42 Angströms. It was concluded that the striations are due to the edge on view of the layers of metal atoms in the soap structure. These striations were still visible after 3½ minute exposure of the soaps to the beam.

## Process

### Manufacture of Lubricating Greases From Organogels of Silica

An improvement in the manufacture of lubricating greases in which the gelling agent is an inorganic colloid is suggested by Carswell and Willcock in U. S. Patent 2,986,518, assigned to Shell Oil Co. The present process consists of gradually adding an organogel of an inorganic colloid in a volatile organic liquid to hot lubricating oil. The temperature and rate of addition are so adjusted that as the gel is added at least 85 per cent of the organic liquid evaporates. The elevated temperature can be maintained after the final addition of the organogel and also vacuum can be employed to remove the final volatile material.

Thus, a silica gel was made by slowly adding a solution of sodium silicate to weak sulfuric acid. After

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repeated washings with water and settling, isopropyl alcohol was used as a wash until the decanted fluid contained less than 2 per cent of water. A surface active agent was then added to this organogel and the latter containing 3 per cent of $SiO_2$ was fed into oil maintained at 100°C. After sufficient gel had been added, the mixture temperature was maintained at 120 to 130°C to remove the final traces of alcohol and finally the mass was milled. With a silica content of 4.5 per cent, a worked penetration of 262 was obtained. Other additives can be included in the lubricant. Also the same method of processing can be used where a synthetic lubricating fluid is employed.

## Composition

### High Temperature Lubricating Greases

According to McGrath and Pellegrini, lubricating greases useful at high temperatures are formed by thickening silicone fluids or diesters with a mixture of a bis-pyrazolone and an organophilic siliceous material. Such products are described in U. S. Patent 2,983,683, assigned to Gulf Research and Development Co. Lubricants are formed by mixing the ingredients at room temperature followed by milling.

A typical lubricating grease was made from 66.4 parts of DC 550 fluid, 30 parts of 4,4′-bis(1-phenyl-2,3-dimethyl-5-pyrazolone) and 3.6 parts of Bentone. This product had a worked penetration of 324, a dropping point of 450+°F., and a performance life of 709 hours in a bearing operating at 400°F. and 10,000 rpm.

### Non-Soap Thickened Lubricating Greases

Odell and Lyons (see U. S. Patent 2,986,517, assigned to Texaco Inc.) find that the addition of 0.5 to 5.0 per cent by weight of formamide to lubricating greases in which the thickener is a solid, such as fine silica or treated clay, improves the yield and stability. The formamide should be present during the homogenization.

For example, a lubricating grease was made by mixing together and milling 15 per cent of fine silica and 85 per cent of a lubricating oil having a viscosity of 310 SUS at 100°F. The product had the following penetrations: unworked 331, worked 60 strokes 380, worked 100,000 strokes 400+. Another mixture containing 15 per cent of the silica, 1.5 per cent of formamide and 83.5 per cent of the oil resulted in a finished product with respective penetrations of 317, 331 and 315.

### Clay-Thickened Lubricating Greases

If the exchangeable inorganic cations of various clays are replaced by certain organo metallic compounds, the treated clays can then be used to produce lubricating greases which have high thermal and oxidation stability as well as resistance to ionizing radiation. Such thickeners are described by Freeman and Peterson in U.S. Patent 2,986,520, assigned to Shell Oil Co.

In the descriptions of various compositions, oxidized organo compounds of iron or chromium are used. For example, a di-n-propyl ferrocene was added to a 2 per cent water slurry of hectorite to give a flocculated organo clay which, after washing, was used to thicken DC 550 fluid. The resulting lubricating grease contained 0.7 per cent di-n-propylferrocene, 6.2 per cent hectorite and 93.1 per cent DC 550. This product had a worked penetration of 249, a water absorption of 35 per cent, and a thin film exposed at 450°F for 24 hours showed a loss of 14 per cent. By using 8 per cent of octadecyl ferricium clay milled into a bright stock, a lubricating grease exhibiting excellent stability when exposed to ionization radiation, was obtained.

### High Temperature Lubricating Greases

By thickening certain lubricating fluids with a mixture of an organo hydantoin and a siliceous material, McCarthy and McGrath (see U.S. Patent 2,979,460, assigned to Gulf

Research and Development Co.) produce lubricating greases which will lubricate anti-friction bearings operating at high rotational speeds and high temperatures.

For example, 63 per cent of G.E. silicone 81717, 33 per cent of 5,5-diphenylhydantoin and 4 per cent of Estersil GT were mixed to a slurry and then given two passes through a colloid mill. The resulting lubricating grease had an unworked penetration of 268, worked penetration of 406, dropping point of 450+°F, and a performance life of 606 hours in a bearing operating at 10,000 rpm and 400°F.

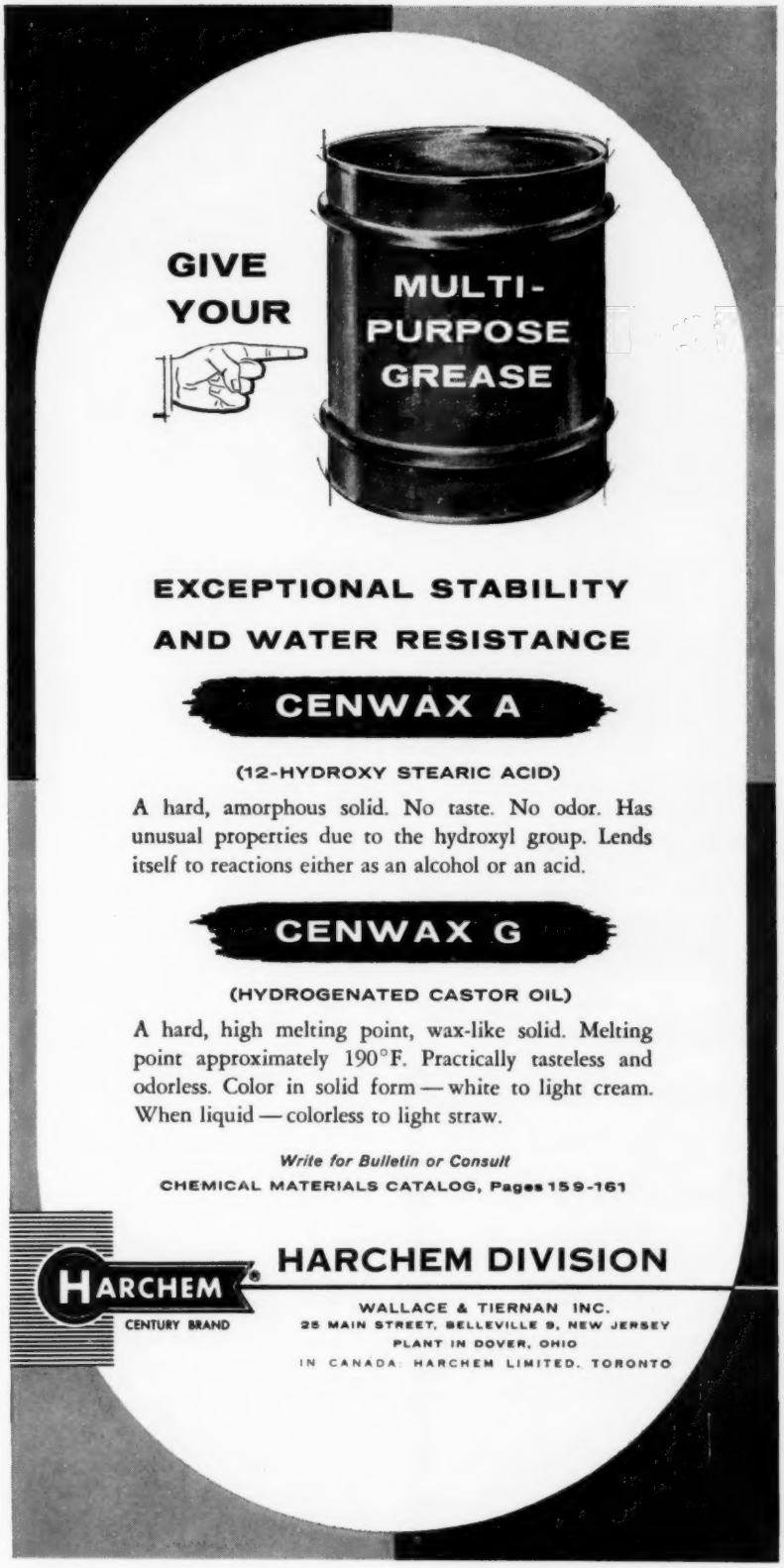
A similar lubricating grease is described in U.S. Patent 2,979,462 by McGrath and Pellegrini where the major portion of the thickener is a spiro hydantoin. A typical product was made by mixing and milling 63 per cent of DC 550 fluid, 33 per cent of spiro (fluorene-9,5′-hydantoin) and 4 per cent of dimethyldicetylammonium bentonite. The resulting lubricating grease had an unworked penetration of 264, a worked penetration of 298, a dropping point of 450+°F, and a performance life in excess of 981 hours in a bearing operating at 10,000 rpm and 400°F.

## Characteristics

**Flow Properties of Lithium Stearate-Oil Model Greases as Functions of Soap Concentration and Temperature. Walter H. Bauer, Alfred P. Finkelstein and Stephen E. Wiberley, ASLE Trans. 3, No. 2, 215-24, 1960.**

Lots of lubricating greases containing 4.2, 8.0 and 12.0 per cent of soap were prepared from lubricating oil thickened with lithium stearate. Another sample contained 11.6 per cent of lithium stearate and 2.8 per cent of stearic acid. The mixtures were heated to 205°C and allowed to cool to ambient temperature undisturbed. No characteristics were given for the resulting lubricating greases.

However, the rate of shear vs. shearing stress was determined on a viscometer in which a low angle cone could be rotated above a tem-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perature controlled plate and in a sample of the lubricating grease. Acceleration and deceleration of the speed of the cone permitted readings which were converted to rate of shear and shear stress. Also, using a constant rate of shear the shearing stress vs. time was determined.

The authors conclude that: "The cone and plate viscometer, with programmed shear acceleration and automatic recording of shear stress vs. shear rate or time, makes possible experimental determinations of yield stresses which are a function of the time scale of the test, and provides a means of measuring initial properties as well as those conditioned by controlled, uniform shear rate and time of shear." Also, "from these flow data, parameters may be determined which are related to the magnitude of the flow resistance due to structural fiber contacts compared to that due to viscous flow of the base oil alone." Equations are suggested for the purpose.

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## Testing

**Scuffing Tests on Gear Oils in the FZG Apparatus. G. Niemann, H. Rettig and G. Lechner. ASLE Trans. 4, No. 1, 71-86 (1961).**

The FZG test is a standardized method in West Germany to evaluate the antiscuffing properties of oils for reduction gears, hypoid gears, automatic transmission gears, etc. The article gives a description of the apparatus and the test procedure.

Since the FZG test is similar to the IAE used in England and the Ryder test used in the United States, a tabulation is given of "Gear Data and Reference Factors" for the three machines.

## Tests

**Antiwear and Extreme Pressure Additives for Greases. S. F. Calhoun, ASLE Trans. 3, No. 2, 208-14, 1960.**

Twenty-nine different additives were evaluated, in proportions of 3, 5 and 10 per cent in some cases and the lower per cent in others, for their antiwear and EP properties when incorporated in a lithium soap-mineral oil lubricating grease. The base grease was made from an 80 viscosity SUS at 100°F oil thickened with preformed lithium stearate. Some mixtures and tests were also made with esters thickened with a modified clay type thickener.

Evaluations were based upon results obtained on the four-ball wear and EP tester, the Falex tester and the Timken tester. A lack of correlation between the various tests was observed.

In spite of this, the following general conclusions were made:

1. Phosphorus-containing compounds tend to contribute antiwear properties to lubricating greases.
2. Sulfur and chlorine compounds exhibit better EP than antiwear qualities with sulfur being somewhat superior to chlorine.
3. Two or more additives can be blended to obtain improvement in antiwear or EP properties.
4. Optimum antiwear and EP qualities seem to depend upon the chemical nature of the compounds rather than upon the chemical element.
5. There is a general tendency for wear to increase as the EP properties are improved.

While the per cent of active elements in the additives is given, the chemical compounds present are not always identified.

**The Performance of Heavily Loaded Oscillating Roller Bearings from 300 F to 600 F. W. A. Glaeser, ASLE Trans. 3, No. 2, 203-7, 1960.**

This investigation was not directed primarily at the performance of lubricating greases, three such products were used in the experiments. These were: at 300 F—MLG 9373; at 450 F—Super Mil ASU-M-40; and at 600 F—High-temperature silicone base grease. No composition or characteristics were given.

It was found that bearings operating without lubricating grease failed in 1/10 to 1/12 the life expected for grease lubricated bearings and that such lubrication was

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*The Literature and Patent Abstracts column is written for NLGI by C. J. Boner, chief research chemist for Battenfeld Grease and Oil of Kansas City, Missouri.*

essential for successful operation at all temperatures. However, it was necessary to replenish the lubricant every 18 hours due to degradation by heat and working.

## Application

**The Effect of Lubricant Viscosity on the Lubrication of Gear Teeth. Ian O. MacConochie and Walter H. Newman, Jr. Wear 4, No. 1, 10-21, (1961).**

A continuous electrical arc was used to measure the film formed between teeth of cast iron gears. During the tests, the load was maintained at a constant value of 36 kg/cm. The lubricant used was an SAE 30 straight mineral oil of paraffinic base. Different viscosities were obtained by varying the temperature of the lubricant supply. Consequently the viscosities tested varied from about 25 to 200 cs.

The oil films between the gear teeth increase in thickness with increase in viscosity. Such films are at a maximum at the pitch line and a minimum at the roots of the teeth. The general conclusions as a result of the investigation are:

(a) The film formed between gear teeth is small and of the order of magnitude of the surface roughnesses and foreign particles in the oil.

(b) At almost all loads the film is hydrodynamic at the pitch line, but for heavily loaded gears lubrication is under boundary conditions or is quasi-hydrodynamic.

(c) That portion of the tooth face operating under boundary conditions depends upon the load, tooth contour, surface finish, impurities in the oil and the lubricant viscosity.

(d) The fact that a hydrodynamic film can be maintained be-

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tween gears in contact under pure rolling action suggests that some other factor is at work, perhaps relaxational effects in the oil.

## Gear Lubrication

**Instantaneous Coefficients of Gear Tooth Friction. G. H. Benedict and B. W. Kelley. ASLE Trans. 4, No. 1, 59-70 (1961).**

It was found that, in a gear contact as simulated on a roller test machine, the instantaneous coefficient of friction follows the concept of transition from boundary to hydrodynamic lubrication. Further, the coefficient was found to increase with increasing load and to decrease with increasing sum velocity, sliding velocity and oil viscosity as each of these qualities varied individually.

The authors point out that the essential difference between rollers and gear teeth is that the tooth profile curvature is continually changing, while in roller tests, the radii and velocities do not change.

Also the investigators agreed: "We do not know from our results the true state of lubrication, whether it be hydrodynamic, elastohydrodynamic, or partial hydrodynamic. In our analysis, we have chosen partial hydrodynamic lubrications as a simple model which helped to visualize the results."

## Theory of Gear Lubrication

**The Load Capacity of Gear Teeth Along the Line of Action. R. R. Stephenson and J. F. Osterle, *Wear* 4, No. 1, 56-63 (1961).**

The hydrodynamic lubrication of involute spur gears is analyzed for an arbitrary point of contact along the line of action. It is found that the maximum load capacity of mating gear teeth does not occur at the pitch point, but approaches this situation for progressively thinner films.

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## Correction

It has been noted that a line of type was dropped in the printing of the article, "Calculating Grease Flow in Pipes," by L. C. Brunstrum and A. W. Sisko, on page 210 of the October, 1961 NLGI SPOKESMAN. The line should have read as follows:

Pressure drop per foot, P/L, is then obtained from

$$\frac{P}{L} = \frac{48\ F}{d}$$

where F is the shear stress.

## Offers Brochure

The theory and use of solid lubricants is thoroughly discussed in a new eight-page, four-color brochure just published by the Alpha-Molykote Corp. Titled "Breaking Lubrication Barriers," the new brochure discusses such barriers to effective lubrication as galling and seizing, extreme pressures, high friction, chemical reaction, temperature and extreme environments, and describes how solid lubricants can overcome them.

The properties of molybdenum disulfide as a dry-film lubricant, including chemical stability, electrical and magnetic properties, thermal stability and coefficients of friction, are presented in detail.

Actual applications describe how solid lubricants have solved difficult lubrication problems. The brochure is illustrated with photographs, charts, graphs and line drawings.

"Breaking Lubrication Barriers," identified as Bulletin 132, may be obtained without cost from the Alpha-Molykote Corp., 65 Harvard Ave., Stamford, Conn.

## IOCA Reelects Officers For Coming Year

The fourteenth annual meeting of the Independent Oil Compounders association saw reelection of officers to a traditional second term. Held Sunday, September 24, at the Villa Moderne Motor hotel, Highland Park, Ill., active members at the meeting reelected Dan Maurin of Midwest Oil, Kansas City, Kans., as president; Paul T. Webster, Jr., Central Petroleum, Cleveland, vice-president; and R. W. Gober, Georgia-Carolina Oil, Macon, Ga., as treasurer.

Herbert M. Hutchins, United Oil, Baltimore . . . John F. McLeod, Mich-I-Penn Oil & Grease, Detroit . . . Bob Petersen, Northland Products, Waterloo, Iowa . . . and A. L. Warden, Warden Oil, Minneapolis, were elected directors. Harry E. Tatman is executive secretary of IOCA.

## Second Edition Available

The second edition of Markley's *Fatty Acids* has been announced by Interscience Publishers, 250 Fifth Ave., New York. Following a first edition which was confined to a treatment of the chemistry of the fatty acids and their physical properties, the second edition has been broadened to include an exhaustive description of the processes for the production of fatty acids and their uses. 782 pages, $27.50

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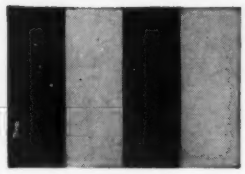

# People in the Industry

## Three Promoted at Fiske

The Fiske Brothers Refining Co. of Newark, N. J., announced three promotions within its executive personnel when Clifford Wolf was named production manager, G. Edward Merkle, Jr. was elevated to plant manager, and Justin P. McCarthy was promoted to the position of plant superintendent.

In making the announcement, Fiske Brothers stated that Mr. Wolf would devote his energies to the improvement of production methods and technical control of manufacture.

The promotions were effective September 22.

## API Names F. N. Ikard Executive Vice-President

Frank M. Porter, president of the American Petroleum Institute, announced that Frank N. Ikard of Wichita Falls, Tex., has been named to the newly-created position of executive vice-president of the Institute.

Mr. Ikard has represented the 13th Congressional district of Texas since 1951.

Born in Henrietta, Tex., he received his bachelor of arts and law degrees from the University of Texas. He served from 1937 to 1948 as a practicing attorney in Wichita Falls, with time out for service in World War II. As an infantryman, he was wounded twice and was later held in a German prisoner of war camp.

He served as a judge in the 30th judicial district court of Texas from 1948 to 1951 before winning a Democratic primary fight for election to Congress. He has run unopposed in five elections since then. Married to the former Jean Hunter of Wichita Falls, he has two sons.

## Du Pont Appoints Diggs

The appointment of Dr. Donald R. Diggs as assistant to the technical manager of Du Pont's petroleum chemicals division has been an-


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nounced by the company.

Dr. Diggs, whose eleven-year career with Du Pont has been in the field of combustion research, will be concerned primarily with following developments relating to the control of automotive exhaust and coordinating the petroleum chemicals division's technical efforts in this area.

A graduate of Northwestern University with a B.S. degree in mechanical engineering, Dr. Diggs worked two years as a research engineer for the National Advisory Committee for Aeronautics. Returning to Northwestern in 1946 as an instructor in mechanical engineering, he was subsequently awarded his M.S. and Ph.D degrees there.

He joined Du Pont in 1950 as an engineer at the petroleum laboratory, Deepwater Point, N. J., and in 1959 was made head of the combustion division there.

## S. A. Ballard Named By Shell Chemical

S. A. Ballard has been named research and development manager for the industrial chemicals division of Shell Chemical Co., it was announced by A. W. Fleer, general manager of Shell's industrial chemicals division.

Mr. Ballard started his Shell career in 1937 as a research chemist for Shell Development Co. in Emeryville, Calif., after completing his doctorate in organic chemistry at Yale university. He held various technical posts there until 1955, when he was appointed associate director of the chemical products department.

In 1957, Mr. Ballard was named director of chemical research at Emeryville. He accepted an administrative position with a Shell affiliated company in The Hague, The Netherlands, in 1958, and held that post until his appointment to this new position.

## Amoco Appoints Christie

William F. Christie has been appointed eastern representative, narket research and development, for Amoco Chemicals Corp., George Rieger, manager of market research and development, has announced.

In his new position Christie will be located in the company's New York office at 555 Fifth Ave., New York City.

Christie has held several positions in the marketing and development departments of Amoco Chemicals since joining the company in 1958. Most recently he was director of market research.

## Climax Names Couch

G. Robert Couch has been appointed technical advisor to the vice-president – eastern operations for Climax Molybdenum Co.

Mr. Couch comes to his new position from the corporate central planning department of American Metal Climax, Inc., the parent company. Previously he had been assistant to the technical director of the Amco division of American Metal Climax, Inc. He joined American Metal Climax in 1957 after fourteen years with National Lead Co., where he was a senior development engineer.

Mr. Couch received his B. S. in Chemical Engineering from Missouri School of Mines and Metallurgy in 1941. He received his M. S. in Chemical Engineering from Newark College of Engineering in 1953 by attending night classes.

# News About NLGI Cont'd

ported slightly poorer results than with the regular 75# can, but none said the can was unacceptable.

*Lightweight Five-Quart Motor Oil Cans*

A check with major suppliers indicates that little work has been done on lighter weights, and there is even less enthusiasm. The subcommittee will put pressure on the suppliers to provide a lighter plate for this can, taking into account the increasing interest being shown in 4-quart cans. (Five companies have now converted to 4-quart cans, with two others contemplating elimination of their 5-quart packages.)

On May 25 American Standards Association sponsored a general conference on Small Containers. Out of this came the appointment of the Packaging Institute as sponsor of the project for "Standardization of terminology, sizes, dimensions and tolerances, methods of measurement, marking and testing of containers having a volume of less than 5 gallons. It is intended that the work initially be confined to containers for petroleum products."

### Meetings and Programs

The next meeting of the Petroleum Packaging Committee will be November 6 and 7 at Detroit in conjunction with the PMMI exhibition, with headquarters at the Pick-Fort Shelby Hotel.

# Industry News

## Qualified Products List For Grease, Rifle

The Rock Island Arsenal, Department of the Army, has announced the intention to establish a Qualified Products List for Grease, Rifle, under Specification MIL - G - 46003 (ORD).

Companies which have a product meeting the requirements of this specification are urged to contact the Commanding Officer, Rock Island Arsenal, Rock Island, Illinois, Attention: Laboratory, for an opportunity to have their products tested.

In making future awards, awards will be made only for such products as have been tested and approved for inclusion in the Qualified Products List.

## Petroleum Packaging Committee to Meet

The Petroleum Packaging Committee will wind up its eleventh year with the forthcoming meeting to be held November 6 and 7, 1961 at the Pick-Fort Shelby hotel in Detroit, Michigan.

This meeting will be held in conjunction with the Packaging Machinery Manufacturer's Institute exhibition.

The PPC business meeting is to be held on November 6, during which time some fifteen subcommittees will report on subjects and standards relative to the packaging, shipping, labeling and handling of the products of the petroleum industry.

The next meeting will be held February 26 and 27, 1962 in New Orleans, La., with headquarters at the Royal Orleans hotel.

## ASTM Officially Changes Its Name

The name of the American Society for Testing Materials was officially changed to the American Society for Testing *AND* Materials with the signing of a court decree on September 18, 1961, amending the society's charter originally granted in 1902 by the Commonwealth of Pennsylvania.

In announcing this change, ASTM President Miles N. Clair said, "The inclusion of the word 'and' in the society's name places added emphasis on the society's research work in seeking knowledge of the nature

If it rolls on an axle or turns in a
bearing or rides on a shaft if it slides
in a groove or moves on a pivot if
it bores or cuts or transmits pressure
one of Sinclair's 500 specialized lubricants
is designed to make it work better. For answers
to your lubrication problems, write today to
SINCLAIR REFINING COMPANY Sinclair
Technical Service Division, 600 Fifth Ave, N.Y. 20 N.Y.

of materials.

"It is a further re-emphasis of one of the basic purposes for which ASTM was founded in 1898. Since then, the work of the society has been devoted to the fulfillment of its basic objectives, unchanged from the original charter which states: 'The corporation is formed for the promotion of knowledge of the materials of engineering, and the standardization of specifications and the methods of testing.' And, to give greater stimulus to the work of the society in the field of materials research, a division of materials sciences was established two years ago."

The change in name was the result of study and discussion with members throughout the country extending over several years. It was also given careful consideration by the ASTM board of directors prior to its approval and adoption by the membership at the annual meeting, June, 1961.

## Technology Spectrum

A technology spectrum showing in detail, areas of research interest and competence at Battelle Memorial Institute is now being offered to those concerned with research and development. Hundreds of types of specialized research are grouped together under ten major areas of interest on this colorful chart. Major areas include biosciences, ceramics, chemical engineering, chemistry, economics, electronics, mechanical engineering, metallurgy, nuclear energy, and physics.

Apart from the specific information it contains, the 16- by 21-inch chart provides a graphic impression of the wide variety of talents and technologies that are drawn together in a modern research center such as Battelle, which annually conducts studies for some 700 different firms and government agencies.

Copies of the chart are available from: Publications Office, Battelle Memorial Institute, 505 King Avenue, Columbus 1, Ohio.

## Bulletin Gives Application Instructions

Typical applications of Molykote G, a grease-type lubricant containing a high concentration of molybdenum disulfide, one of the most effective lubricants known for extreme pressure applications, are featured in a new illustrated bulletin just published by the Alpha-Molykote Corp. The ability of the material to prevent galling, seizing, fretting and metal pick-up is described.

Such actual applications as lubrication of heavy machine ways and guides, prevention of galling and seizing in threaded connections, reduction of cam wear, reduction of metal pick-up on lathe centers and steady rests, press fitting of bearings and gears on shafts, and prevention of fretting on splines are discussed and illustrated.

The four-color, two-page bulletin describes the lubricating characteristics of Molykote G and gives application instructions. Bulletin 131 is available without cost from the Alpha-Molykote Corp., 65 Harvard Ave., Stamford, Conn.

## Bardahl Offers Booklet

Bardahl now offers an industrial publication dealing with lubricating grease, written to serve both management personnel and lubrication specialists.

Offered free to interested parties, the 32-page booklet covers the basic principles, properties, types and rules for choice and application of lubricating grease, including the effect of additives on the properties of grease.

For the booklet write Bardahl, 1400 N. W. 52nd St., Seattle 7, Wash.

## New Plant Facilities

Southwest Grease & Oil Co., Wichita, Kans., has announced the completion of a $750,000 rebuilding program covering manufacturing facilities, filling equipment, finished products storage tanks and warehouses. Mr. H. A. Mayor, Sr., Southwest president, described the new plant as, "A completely new concept in grease plant operation. These new facilities, combined with Southwest's newly-developed 'Electro-Thermatic'® process, give our operation flexibility and production capacity never before achievable. One of the greatest benefits of these new facilities is that tolerances on critical specification lubricants can be held to a degree never before possible."

A few of the interesting statistics on the new facilities incorporated into the modernization program:

Twenty-one miles of new piping to prevent any possibility of "in line" product contamination.

Finished product storage tanks holding over 3,000,000 pounds of finished products to assure prompt delivery of specification lubricants and to level production loads.

Manufacturing facilities expanded to meet increasing demand; and capable of producing 750,000 pounds of lubricants daily.

**The C. W. Nofsinger Company**
CONSULTING ENGINEERS
Grease Plants
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Fine Lubricating Qualities For Greases and Fluid Lubricants
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**Comparison of Lithium 12-hydroxystearate greases produced by conventional open kettle and by Contactor**

**NLGI GRADE 1**

Conventional

**OPEN KETTLE** Max. Proc. Temp. 330° F.

Hydrogenated Castor Oil 10.50% Wt.

Lithium Hydroxide, monohydrate 1.57% Wt.

Mineral Oil* 87.93% Wt.

CUT COSTS FROM THIS

**7¢** PER LB. INGREDIENT COST

Modern

**STRATCO CONTACTOR** Max. Proc. Temp. 400° F.

Hydrogenated Castor Oil 6.00% Wt.

Lithium Hydroxide, monohydrate 0.90% Wt.

Mineral Oil* 93.10% Wt.

TO THIS WITH STRATCO

**5.8¢** PER LB. INGREDIENT COST

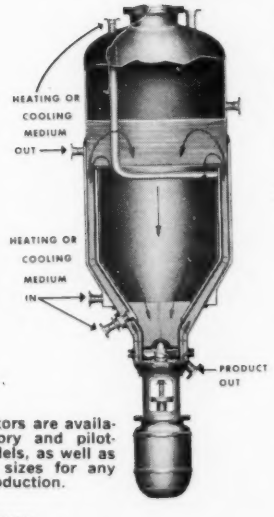
*95 V.I. Solvent Treated Mid-Cont. Neutral. 300 SSU @ 100° F. Oil used.

# STRATCO CONTACTORS

## SAVE 17%* on Grease Ingredient Costs!

Stratco Contactors provide continuous or batch mixing with very short time cycles, less soap, less laboratory control. Intimate contact between reactants plus close control of temperature makes it possible to produce a better product while using less of the more expensive ingredients. Savings average approximately 17% . . . a very substantial contribution to your profit margin!

*NLGI Grade 1 used in this example. Comparable savings can be demonstrated in manufacture of Grades 2 and 3.




Stratco Contactors are available in laboratory and pilot-production models, as well as in a range of sizes for any commercial production.

**TESTS ON FINISHED GREASES**

| | OPEN KETTLE | CONTACTOR |
|---|---|---|
| PENETRATION | | |
| Unworked | 341 | 319 |
| Worked, 60 Strokes | 333 | 314 |
| Worked, 100,000 Strokes | 339 | 363 |
| DROPPING POINT, °F | 380 | 388 |
| WHEEL BEARING TEST | | |
| ASTM D-1263 @ 250° F | Pass | Pass |
| Leakage, gms. | 3.6 | 4.0 |
| WATER WASHOUT TEST | | |
| ASTM D-1264 @ Room Temp., Loss, Wt. % | 5.2 | 1.38 |

REPRESENTATIVES

D. D. Foster Co., Pittsburgh, Pa.
D. D. Foster Co., S. Charleston, W. Va.
The Rawson Co., Inc., Baton Rouge
F. J. McConnell Co., New York
Lester Oberholtz, Los Angeles
Rawson-Houlihan Co., Inc., Houston
Rawson-Houlihan Co., Inc., Beaumont, Texas

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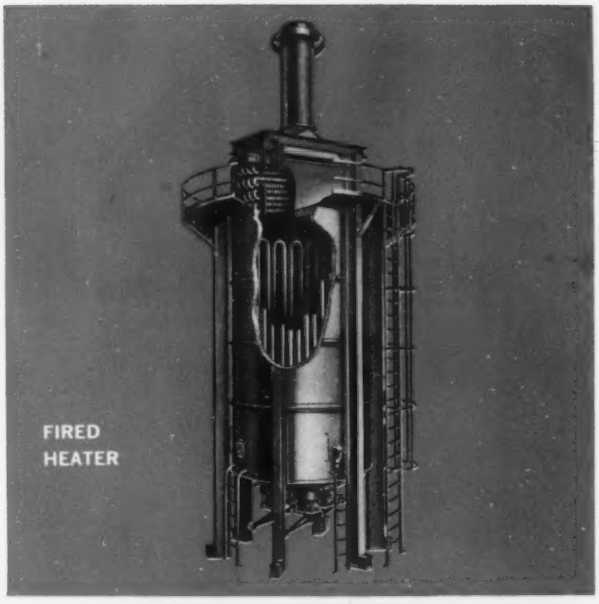
G1161

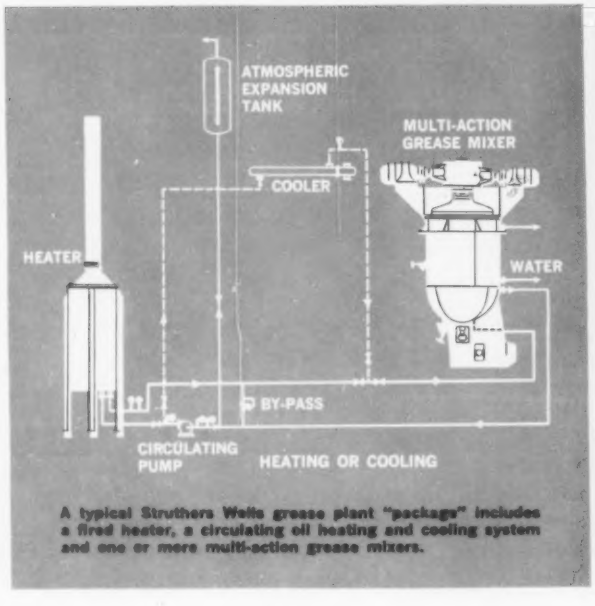
**4601 Madison Ave. Kansas City 12, Mo.**

for maximum
grease plant efficiency...

# A COMPLETE PACKAGE

engineered by
**STRUTHERS WELLS**




A typical Struthers Wells grease plant "package" includes a fired heater, a circulating oil heating and cooling system and one or more multi-action grease mixers.

Accomplishing peak efficiency in your grease plant necessitates a carefully engineered "complete package" including a fired heater, a mixer and a cooling system. The requirements of each plant are reviewed by Struthers Wells engineers. A hot oil furnace which is direct fired with gas or oil may be selected, or it may be electrically heated. The heater may be sized for one grease kettle or a whole battery of kettles. In each case, our engineers will recommend the design that will provide maximum economy, reduced production time and uniformly high product quality. Ask for technical treatise on "Grease Mixer Design" which gives more details.

Struthers Wells

**STRUTHERS WELLS CORPORATION**

WARREN, PA.

Plants in Titusville, Pa. and Warren, Pa.